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ANALYTICAL ABSTRACTS

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

430. **Unconscious individual anomalies of estimation during weighing, and their effect on the exactness of micro analyses.** H. Gysel (*Mikrochim. Acta*, 1953, [3], 266–282).—Errors are introduced into micro-analytical work in estimating readings between scale divisions of microbalances. Apparently there is an unconscious preference for certain numbers (0, 2, 5 and 8) and rejection of the others (1, 3, 4, 6, 7 and 9) that is characteristic of each operator. Errors due to this psychological factor may amount to 0·25 per cent. in microchemical C and H determinations. Techniques for eliminating these errors are discussed. It is concluded that further development in micro-analysis must take into account this phenomenon, for instance, in the provision of improved balances. J. H. WATON

431. **Development, present state and prospects of organic spot test analysis.** F. Feigl (*Mikrochim. Acta*, 1953, [3], 157–177).—A review of some of the problems encountered in the development of organic spot test analysis, and of some of the techniques of that subject.

The paper forms chapter I of vol. II (Fourth Edition) of the author's "Spot Test Analysis," translated by R. E. Oesper. J. H. WATON

432. **Preparation of ammoniacal cuprous chloride.** B. B. Bach, J. V. Dawson and L. W. L. Smith (*Chem. & Ind.*, 1953, [48], 1279–1280).—The preparation is described of ammoniacal cuprous chloride reagent in which the amount of cupric salt is very small. Cu_2Cl_2, in an atmosphere of N, is extracted repeatedly with 5 per cent. HCl to remove cupric salt and then dissolved in aq. NH_4Cl and conc. aq. NH_3. The resulting reagent is kept under N, and gives complete absorption of CO.
D. BAILEY

433. **Critical relative humidities of some salts.** W. E. Windsor, F. Sobel, V. B. Morris, jun., and M. V. Hooper (*Rev. Sci. Instrum.*, 1953, **24** [4], 334).—The critical relative humidities (the relative humidity which exists in equilibrium with a saturated solution of a salt) for 21 salts at 25° C are listed. G. SKIRROW

434. **An isopiestic method for the micro-determination of molecular weights.** J. E. Morton, A. D. Campbell and T. S. Ma (*Analyst*, 1953, **78**, 722–725).—A simple method for determination of the mol. wt. of non-volatile compounds is based on the isopiestic method of Sinclair for determination of vapour pressures (*Brit. Abstr. A*, 1933, 587; and 1934, 1173). A soln. of the compound in a volatile solvent is rocked at constant temp. in a desiccator with a soln. in the same solvent of a substance of known mol. wt. When the two soln. are isopiestic the mol. wt. of the compound is calculated from that of the standard substance and the concn. of the two soln. Concordant results were obtained with 3- to 7-mg samples in a number of solvents. A. O. JONES

435. **Acetylation of paper for use in chromatography.** H. S. Burton (*Chem. & Ind.*, 1953, [46], 1229–1230).—Whatman No. 1 filter-paper is immersed first in 10 per cent. $HClO_4$ for 45 min. and then, after blotting, in a mixture of 150 parts of acetic anhydride and 350 parts of benzene for 75 min. at 0° C. The paper is then washed with water and dried. The fibres have modified properties as regards their affinity towards adsorbates and retention of the stationary phase in a two-phase system. E. G. BRICKELL

2.—INORGANIC ANALYSIS

436. **The determination of small amounts of potassium, calcium and magnesium in sodium and its compounds.** L. Silverman and K. Trego (*Analyst*, 1953, **78**, 717–721).—Metallic Na, NaOH and Na salts are converted to chloride, the bulk of which is then pptd. by treating the saturated soln. with dry HCl gas. The NaCl collected in a sintered-glass crucible is washed with HCl and discarded. The filtrate and washings are evaporated to dryness, treated with HNO_3 and $HClO_4$ and then with ethyl acetate to remove residual $NaClO_4$, and K is determined in the usual way. For sequence determination of Ca, Mg and K, the procedure is the same up to the stage before addition of HNO_3 and $HClO_4$. Water is added, and, after adjustment of the pH to between 9·5 and 10·5 with aq. NH_3, Ca and Mg are pptd. together as 8-hydroxyquinolates, the filtrate being reserved for determination of K. After treatment of the ppt. with HNO_3 and $HClO_4$ to destroy the complex, Ca is pptd. as oxalate and Mg is separated from the filtrate as 8-hydroxyquinolate. A. O. JONES

437. **The colorimetric estimation of caesium.** C. Duval and M. Doan (*Mikrochim. Acta*, 1953, [3], 200–211).—Ten colorimetric estimations are described for Cs separated from K and Rb by filter-paper chromatography. The methods are those of flame spectrography of the chloride, dipicrylaminate and picrate; colorimetric estimation of the dipicrylaminate, picrate and platinichloride (with 2 modifications); pptn. as $Cs_3[Co(NO_2)_6].H_2O$, followed by the colorimetric determination of the Co with nitroso-R salt or thiocyanate, or of the NO_2' by Griess's method or the modification with procaine. The methods appear to have approx. the same precision. The method of flame spectrography does not demand previous separation of Cs; it is rapid but costly. It is best confined to CsCl and $CsNO_3$, and the use of organic compounds of Cs is best avoided. The modified version of Griess's method is preferred, in which the NO_2' of the $Cs_3Co(NO_2)_6$ complex is determined colorimetrically.
J. H. WATON

438. **Electrolytic determination of copper with an isolated anode.** D. G. Foster (*Anal. Chem.*, 1953, **25** [10], 1557–1558).—The method is applicable to the determination of Cu in brass. The sample is dissolved in 15 ml dil. HCl (2 + 1) with the aid of 5 ml of 30 per cent. H_2O_2, the excess being removed by boiling, and 1 to 2 g of urea are added. Anolyte (135 ml of HCl and 14 g of hydrazine dihydrochloride per litre) is added, and the weighed cathode is clamped into place. Electrolysis is started with the control set at −0·22 V *vs.* the S.C.E., mechanical stirring taking place throughout the operation. Automatic electrolysis control is applied. The method is accurate and precise to within 2 parts per 1000 for determination of Cu in brass containing 1 to 20 per cent. of Pb. Separation from Pb is satisfactory provided the control potential does not exceed − 0·30 V *vs.* the S.C.E.

G. P. Cook

439. **2:2-Dilepidine as spectrophotometric reagent for copper.** J. Gillis, J. Hoste and Y. Van Moffaert (*Meded. Vlaamsche Acad. Kl. Wet.*, 1953, **15**, No. 7, 12 pp.).—The reagent is a 22 per cent. soln. of 4:4′-dimethyl-2:2′-diquinolyl (m.p. 253°–255° C), prepared by catalytic dehydrogenation and condensation of dimethylquinoline at 390° C for 24 hr. This forms a Cu complex having a mol. extinction coeff. of 7020, which is 12·7 per cent. > that of the complex with 2:2′-diquinolyl ("cuproin"), and having a max. at 548–550 mμ; its solubility in *iso*pentanol (0·02 per cent.) and partition function between water and *iso*pentanol (828) are both < those of cuproin. The Cu complexes formed with the above reagents possess, in comparison with those formed by 1:10-phenanthroline and its 2:9-dimethyl and 2:4:7:9-tetramethyl deriv., the advantage of stability towards atm. oxygen, although their extinction coeff. are somewhat lower.

P. S. Arup

440. **Colorimetric estimation of copper using rubeanic acid.** P. B. Janardhanan (*J. Sci. Ind. Res., B, India*, 1953, **12** [11], 514–517).—Concn. of Cu not exceeding 5 p.p.m. may be accurately determined by measuring the intensity at 390mμ of the green Cu - rubeanic acid complex formed at pH 3–4 in soln. containing 0·5 ml of 0·5 per cent. rubeanic acid in ethanol and 1 ml of 1 per cent. aq. gum arabic or gelatin as stabiliser. As rubeanic acid shows strong absorption at 390mμ, the vol. of reagent added to the sample should equal that added to the blank. For higher concn. of Cu, or in presence of Fe···, red filters (600–660 mμ) should be used; citrate buffers do not eliminate interference due to Fe···. Since the colour obeys Beer's law, small concn. of Cu, *e.g.*, in industrial waters, can be calculated from the optical densities of the sample, a known standard and a blank in the region of 390mμ.

W. J. Baker

441. **The determination and distribution of copper in sea water.** T. J. Chow (*Dissert. Abstr.*, 1953, **13** [5], 661).—The use of sodium diethyldithiocarbamate instead of dithizone as the colour forming agent in the spectrophotometric determination of Cu in sea water provides higher sensitivity, greater accuracy, simpler operation and reduces interference of other ions. Xylene is used to extract the Cu diethyldithiocarbamate. The method has been used in the analysis of many water and sediment samples; the distribution and fluctuation of Cu concn. in sea water has been studied also.

N. M. Waller

442. **The quantitative separation of copper, lead and tin by cathodic deposition.** G. H. Aylward and A. Bryson (*Analyst*, 1953, **78**, 651–655).—The quantitative separation of Cu and Pb from Sn by cathodic deposition from H_3PO_4 solution is described. Sn forms an anionic complex that is not reduced at the cathode, and Cu is separated from Pb by deposition at a controlled potential. After the electrode with its copper deposit has been weighed, it is returned to the cell and the Pb is deposited upon it in metallic form. Sn is determined iodimetrically after reduction with Ni. Optimum working conditions have been established and the method is applicable to determination of the three metals in copper-based alloys and of Cu and Sn in white metal.

A. O. Jones

443. **Diethylammonium diethyldithiocarbamate for the separation and determination of small amounts of metals. I. The successive determination of small amounts of copper, manganese and iron in organic compounds.** P. F. Wyatt (*Analyst*, 1953, **78**, 656–661).—In the scheme described Fe is isolated as cupferrate and Cu and Mn separately as diethyldithiocarbamates. The three elements are determined absorptiometrically as their coloured diethyldithiocarbamates in $CHCl_3$ solution. Fe may be determined, somewhat less satisfactorily, by measurement of the colour of the cupferron solution. Ni and Co interfere with the manganese determination but the method described can be used for the separation of Mn, the determination being then completed by oxidation with KIO_4 and absorptiometric measurement of the $KMnO_4$. A modification of the procedure for Cu is necessary in presence of Bi.

A. O. Jones

444. **Semimicro determination of beryllium in copper - beryllium alloy.** H. Gotô and Y. Kakita (*Sci. Rep. Res. Inst. Tohoku Univ., A*, 1953, **5** [2], 163–171).—The development of a method of determining Be in small samples of Cu - Be alloys is described. After separation by precipitation with guanidine carbonate, the Be is determined colorimetrically with aluminon (aurinetricarboxylic acid). Results obtained on five different samples are tabulated.

G. C. Jones

445. **Purity tests. VIII. Limit test for magnesium.** H. B. Lauritsen (*Dansk Tidsskr. Farm.*, 1953, **27** [11], 241–251).—Polyhydroxy alcohols limit interference by Fe··· and Zn··· in the titan-yellow test for Mg. The following test is based on this observation, and on that by Reimers and Gottlieb ("Limit Tests for Impurities," Danish Pharmacopoeia Commission, Vol. I, Copenhagen, 1946) that interference by Zn·· or Al··· is limited by oxalate ions. To 9 ml of the test soln. are added 1 ml of glycerol (freed from Mg if necessary by redistillation), 3 drops of aq. 0·05 per cent. titan yellow, 5 drops of 0·5 *N* NH_4 oxalate and 5 ml of 2 *N* NaOH. The coloration (stable during 60 min.) is compared photometrically, by use of a wide spectral range (Pulfrich filter S53), with that produced by a standard containing 0·5 μg of Mg per ml. Beer's law is not followed. Slight corrections are applicable for test soln. containing 10 μg of Fe··· or Zn·· per ml.

P. S. Arup

446. **The indirect polarographic determination of calcium by chloranilic acid.** B. Breyer and J. McPhillips (*Analyst*, 1953, **78**, 666–669).—In the polarographic method described, Ca is pptd. quantitatively by chloranilic acid (prep. described)

and determined without removal of the ppt. by measurement of the diffusion current of the residual chloranilic acid, the value obtained being referred to a calibration graph. Co, Zn, Pb, Mn, Cd, Ni, Al, Cu and Ag ions interfere by reacting with the reagent. Fe^{III}, Hg^{II}, Mg, Li, Ba, Cr and Sr do not interfere below specified concn. Na, K or NH_4 ions do not interfere. The method has been applied to determination of Ca in blood serum and in milk

A. O. JONES

447. **Rapid precipitation of barium sulphate.** Robert B. Fischer and T. B. Rhinehammer (*Anal. Chem.*, 1953, **25** [10], 1544–1548).—Investigation of the influence of various conditions upon the morphology and particle size of pptd. $BaSO_4$ led to the development of the following rapid procedure for SO_4'' determination. The pH of the SO_4'' soln. is adjusted to 1·0 with HCl and the soln. is heated to 95° C. $BaCl_2$ (0·02 *M*) at 95° C is poured rapidly into the sulphate soln., an initial 10 to 20 per cent. excess being added. The soln. is stirred manually for 2 to 3 min., cooled and filtered. The ppt. is washed, ignited and weighed. The sulphate soln. should be 0·006 to 0·16 *M* and the total vol. 150 to 200 ml. If alkali salts are present in high concn. the pH is altered to 5·0. Results obtained by the method are within 1 part per 1000 for accuracy and precision. The only advantage of this method over conventional procedures is greater speed and ease of handling. Interferences are the same. A detailed description of the influence of conditions on the size of $BaSO_4$ particles is given. G. P. COOK

448. **The evaluation of barium sulphate precipitates by a titrimetric method.** R. Belcher, D. Gibbons and T. S. West (*Chem. & Ind.*, 1954, [5], 127–128).—The method depends on dissolving $BaSO_4$ in an excess of ethylenediaminetetra-acetic acid (**I**) and titrating the soln. with standard aq. $MgCl_2$. The $BaSO_4$ is pptd. by the method of Kolthoff and Sandell ("Textbook of Quantitative Inorganic Analysis," Macmillan, 1943), and a paper-pulp pad is used for filtration; the well-washed pad and ppt. are transferred to the original flask and a known two-fold excess of 0·02 *M* soln. of **I** is added, followed by 5 ml of 9 *M* aq. NH_3. The mixture is boiled (5 to 10 min.) and cooled, 10 drops of freshly-prepared 0·5 per cent. Solochrome Black in alcohol are added, and the solution is titrated with aq. 0·02 *M* $MgCl_2$ to a clear red end-point (1 ml of 0·02 *M* **I** = 0·6412 mg of S). Fe and other metals forming complexes with **I** should be removed by re-pptn. of the $BaSO_4$ by the Přibil - Maričová method. Accuracy is high for SO_4'' and $Ba^{··}$.

W. J. BAKER

449. **The determination of zinc by titration with disodium ethylenediaminetetra-acetate.** N. Strafford (*Analyst*, 1953, **78**, 733–734).—The amperometric titration of Zn with the disodium salt of ethylenediaminetetra-acetate (**I**) has been described (Pickles *et al.*, *Brit. Abstr. C*, 1953, 339). The same micro titration can be made to a visual end-point by the method of Biedermann *et al.* (*Brit. Abstr. C*, 1949, 4) with Solochrome Black WDFA as indicator. Zn ions (0·2 mg in a vol. of 10 to 15 ml) can be titrated with a 0·01 *M* soln. of **I** in presence of an NH_4Cl - aq. NH_3 buffer by means of a 0·5 ml micrometer syringe burette to an accuracy of within ± 2 μg. Presence of Ca can be masked completely by addition of NaF (Pickles *et al.*, *loc. cit.*).

A. O. JONES

450. **Preparation of a material rich in "cadion" suitable for detection of cadmium.** E. Fisher B. T. Estes and J. E. Rose (*Analyst*, 1953, **78**, 729–730).—A simple method for preparing a crude *p*-nitrodiazoaminobenzene by diazotisation of a mixture of *p*-nitroaniline and aniline is described. This crude product is rich in *p*-nitrodiazoaminoazobenzene, the "cadion" of Dwyer (*Brit. Abstr. A I*, 1937, 262; *Brit. Abstr. A II* 1937, 144; and 1938, 318) and, in alcoholic solution, serves as a reagent for detecting cadmium.

A. O. JONES

451. **Determination of borate in presence of silver.** S. Z. Haider (*Analyst*, 1953, **78**, 673–675).—A modification of the usual method for determination of BO_3''' in presence of $Ag^{·}$ is described. The Ag is converted to AgCl by addition of HCl (NaCl if H_3BO_3 is present) and the AgCl is converted into a soluble complex by addition of controlled amounts of HCl and $Na_2S_2O_3$. After neutralisation to methyl red and addition of glycerol the liquid is titrated to the phenolphthalein end-point as usual.

A. O. JONES

452. **Analysis for industry. [Analytical methods for aluminium. I.]** M. Kapel (*Ind. Chem.*, 1953, **29**, 539–541).—This first part deals with the determination of Al. 62 references.

I. J. METCALFE

453. **Analysis for industry. [Analytical methods for aluminium. II.]** M. Kapel (*Ind. Chem.*, 1953, **29**, 573–576).—This second part of a review of published methods has 86 references.

BRITISH ABSTRACT

454. **Micro-analysis of silicate rocks. IV. Determination of alumina.** C. C. Miller and R. A. Chalmers (*Analyst*, 1953, **78**, 686–694).—In the procedure described, SiO_2 is removed by heating with HF and H_2SO_4 and the residue is then fused with $KHSO_4$ and dissolved in HCl. Fe, Ti, Va and Zr are removed by means of cupferron with *o*-dichlorobenzene as organic solvent. The aq. soln. is treated with acetylacetone under specified conditions and the Al and Be acetylacetonates are extracted with ether. After transference of the Al and Be complexes into HCl, the Al alone is pptd. and weighed as the 8-hydroxyquinolate. The method has been applied in presence of all the elements commonly found in silicate rocks.

A. O. JONES

455. **Use of nitrilotriacetic acid for separation of ceric earths by ion exchange.** J. Loriers and D. Carminati (*Compt. Rend.*, 1953, **237** [21], 1328–1330).—La, Nd and Pr may be almost completely separated from Ce and Sm in large samples (10–300 g) of rare earths by one continuous elution with nitrilotriacetic acid (10 to 30 g per litre) at pH 4 to 6 by means of acidified Dowex-50 as ion-exchanger in a column 130 cm long by 3 cm diameter. Efficient extraction and separation of La, Nd and Pr by the eluting soln. depends on a reduced concn. of $H^{·}$ and an increased concn. of $NH_4^{·}$ and of nitrilotriacetic acid (1 to 2·5 per cent.).

W. J. BAKER

456. **Determination of carbon in aluminium metals.** H. Gotô and S. Takeyama (*Sci. Rep. Res. Inst. Tôhoku Univ., A*, 1953, **5** [2], 159–162).—Determination of total carbon by direct combustion of Al was not possible. In the method subsequently developed the combined carbon was determined from the gas evolved on dissolving the Al in $FeCl_3$ soln. The apparatus used is illustrated. The graphitic carbon was determined by normal combustion of the residue. The results for three

determinations on each of seven different Al samples are tabulated. The method could be applied to metals other than Al. G. C. JONES

457. **Determination of germanium with the help of a radioactive germanium isotope.** L. K. Bradacs, I.-M. Ladenbauer and F. Hecht (*Mikrochim. Acta*, 1953, [3], 229–243).—By means of the radioisotope ^{71}Ge, the determination of Ge as the oxine germanomolybdate is found to be quant. When ^{71}Ge is used in various methods for determining small amounts of Ge in minerals such as Zn blende, it is found that the pptn. with H_2S in H_2SO_4 and the distillation in Cl are quant. However, pptn. with H_2S from HCl soln. and soln. of the sulphide ppt. in aq. NH_3 are not quant., and greatest losses occur in dissolving GeO_2 in hot H_2O. The radioactive isotope procedure is also applied to the determination of Ge in a coal ash. J. H. WATON

458. **Siloxene as a chemiluminescent indicator in chromate titration for determination of lead.** F. Kenny and R. B. Kurtz (*Anal. Chem.*, 1953, **25** [10], 1550–1551).—Siloxene can be used as a chemiluminescent indicator in the volumetric determination of Pb if a multiplier photometer is used. In order that the indicator will emit light at or near the stoicheiometric point the pH should be between 1·9 and 3·0; it should be adjusted before addition of the siloxene. Variation in the amount of indicator between 0·02 and 0·08 g has no appreciable effect on the end-point. The accuracy of the method under these conditions of pH and with this amount of indicator is 0·7 parts per 1000 with a precision (average deviation of a single observation) of 1·3 parts per 1000. Mn^{II}, Ni^{II}, Fe^{III}, Zn^{II}, Ca^{II} and Mg^{II} interfere to the extent of 3 parts per 1000 or less. G. P. COOK

459. **On the formation of complex ions applied in analytical chemistry. VI. Studies on complexes of lead and cadmium malonates.** S. Suzuki (*Sci. Rep. Res. Inst. Tôhoku Univ., A*, 1953, **5** [2], 147–152).—As a continuation of this series, the complexes of lead and cadmium malonates were measured at 25° C by a potentiometric compensation method involving an ion concentration cell and quinhydrone electrode. The method has been described previously (Suzuki, *Sci. Rep. Res. Inst. Tôhoku Univ.*, 1951, **3**, 292; 1952, **4**, 176 and 464; 1953, **5**, 16 and 47). Details of the reagents and cells used are given and the experimental results are tabulated. The following values were obtained: for lead malonate, $K = 4{\cdot}49 \times 10^{-5}$; for Cd malonate, $K = 7{\cdot}14 \times 10^{-5}$. Dissociation degrees were also calculated. G. C. JONES

460. **On the formation of complex ions applied in analytical chemistry. VII. Studies on complexes of silver citrate and lead acetate.** S. Suzuki (*Sci. Rep. Res. Inst. Tôhoku Univ., A*, 1953, **5** [2], 153–158).—As a continuation of this series, the complexes of silver citrate and lead acetate were measured at 25° C, by a potentiometric compensation method, by means of an ion concentration cell and quinhydrone electrode. The method has been described elsewhere. Details of the reagents and cells used are given and the experimental results are tabulated. The following values were obtained: for Ag citrate, $K = 1{\cdot}14 \times 10^{-3}$; for Pb acetate, $K = 1{\cdot}45 \times 10^{-4}$. Dissociation degrees were also calculated. G. C. JONES

461. **Precipitation of iodates from homogeneous solution. Separation of thorium iodate.** C. R. Stine and Louis Gordon (*Anal. Chem.*, 1953, **25** [10], 1519–1522).—A method for the formation of IO_3' in homogeneous solution is described. The IO_3' is formed by the reduction of IO_4' with ethylene glycol, which is produced by the hydrolysis of 2-hydroxyethyl acetate. Several insoluble iodates can be pptd. in a dense and easily filterable form. In the application to Th, the solution of the salt is concentrated and made 2·5 to 3·0 *N* with HNO_3. $NaIO_4$ is added, followed by 2-hydroxyethyl acetate, mechanical stirring being applied throughout the pptn. stage. The ppt. is then washed by decantation with a soln. of 0·8 per cent. KIO_3 in 10 per cent. HNO_3 and finally dissolved in 10 per cent. HCl. This soln. is evaporated to dryness and boiled for 1 hr. at 110° C. The residue is heated with 50 ml of 10 per cent. HCl and the undissolved SiO_2 is filtered off. The Th can then be determined by the usual methods. A double pptn. effects quant. separation of the Th from large amounts of rare earths and phosphates. Other interfering elements are Fe^{III}, Ti^{IV} and Zr. A detailed method for the determination of Th in monazite sand is also given. G. P. COOK

462. **Colorimetric and volumetric estimation of thorium with oxalohydroxamic acid.** S. K. Dhar and A. K. Das Gupta (*J. Sci. Ind. Res., B, India*, 1953, **12** [11], 518–520).—Small amounts (0·025–0·25 μg) of Th are quant. precipitated as oxalohydroxamate $[Th_5(OH)_2.(C_2N_2O_4H_2)_9.xH_2O]$ by means of hot 1 per cent. aq. oxalohydroxamic acid in presence of NH_4Cl and NH_3 at pH 7 to 9. The ppt. is dissolved in dil. acetic acid and the hydroxamic acid is determined colorimetrically (with a 500-mμ filter) as the ferric-hydroxamic acid complex formed after addition of a slight excess of $FeCl_3$. The relation between optical density of the complex and Th concn. is linear; a min. of 1 p.p.m. of Th can be determined. In the micro-volumetric method, the basic Th oxalohydroxamate is hydrolysed with boiling 6 *N* HCl, the liberated NH_2OH is treated with excess of aq. 0·02 or 0·1 *N* $TiCl_3$ and the soln. is then back-titrated with aq. 0·02 or 0·1 *N* ferric alum. A min. of 1 mg of Th can be accurately determined. W. J. BAKER

463. **Tests for nitrite and nitrate applicable over wide concentration ranges.** P. Woodward (*Analyst*, 1953, **78**, 727–729).—The neutral nitrite soln. (1 ml) is treated with 0·5 ml of sulphanilic acid solution (1 per cent. w/v in 5 *N* acetic acid) and, after diazotisation has proceeded for 10 sec., the mixture is poured into 2 ml of a fresh soln. (≃ 0·5 per cent.) of 1-naphthol in 2 *N* NaOH (or aq. NH_3). SO_4'', NO_3', F', Cl', Br', SiO_3'', BO_3''', PO_4'''', AsO_4''' and the common organic acids do not interfere. I', CrO_4'', SO_3'' and S_2O_3'' interfere but Pb acetate may be added to ppt. them and the test is then made without removal of the ppt. CN' and CNS' interfere when present in large amounts but they can be removed by controlled addition of Ag_2SO_4. For identification, nitrate is reduced to nitrite by Zn dust under specified pH conditions. If NO_3' is present as well as NO_2' it is removed by boiling the neutral soln. with NH_4Cl. A. O. JONES

464. **The separation of phosphorus and iron by means of cation exchange resin.** Y. Yoshino (*Bull. Chem. Soc. Japan*, 1953, **26** [7], 401–403).—Provided that Fe is reduced to the ferrous state with SO_2, the separation of PO_4'''' from Fe''' by means of

a cation-exchange resin is quant. Without reduction, and with the help of radioactive ^{32}P, it is shown that the separation of PO_4''' is increasingly poorer for diminishing values of the P to Fe ratio. The distribution of PO_4''' on the resin column when 1 millimole of $FeCl_3$ and 1 millimole of phosphoric acid is taken is examined both with an autoradiograph and by high-frequency measurements; it is concluded that the PO_4''' is held on the column by direct interaction with the Fe···. J. H. WATON

465. **The photometric determination of phosphorus in copper-based alloys containing tin.** H. K. Lutwak (*Analyst*, 1953, **78**, 661–665).—The method of the A.S.T.M. (E62–50T) for determination of P in copper-based alloys was examined to obviate certain shortcomings. The sensitivity of the phosphovanadomolybdate complex to acidity and dilution was examined and optimum conditions for colour development were established. Hence calibration graphs obeying Beer's law were drawn. Acid concn. is arranged to prevent separation of metastannic acid. Calibration graphs are prepared by the same procedure for alloys of low or of high P content. A. O. JONES

466. **Separation of several inorganic compounds of phosphorus and arsenic by paper chromatography.** Y. Volmar, J. P. Ebel and Y. Fawzi Bassili (*Bull. Soc. Chim. France*, 1953, **20** [11–12], 1085–1088).—Two acid solvents: *iso*propanol (75 ml) - water (25 ml) - trichloroacetic acid (5 g) - 20 per cent. w/v aq. NH_3 (0·3 ml) (**I**) and ethanol (80 ml) - water (20 ml) - trichloroacetic acid (5 g) - 20 per cent. w/v aq. NH_3 (0·3 ml) (**II**), and two alkaline solvents: *iso*propanol (40 ml) - *iso*butanol (20 ml) - water (39 ml) - 20 per cent. w/v aq. NH_3 (1 ml) (**III**) and ethanol (30 ml) - *iso*butanol (30 ml) - water (39 ml) - 20 per cent. w/v aq. NH_3 (1 ml) (**IV**) are used for the paper chromatography of 5–20 μg of P as hypophosphite, phosphite, pyrophosphite, hypo-, ortho- and pyro-phosphates. R_F values are quoted for these ions in solvents **II** and **IV**. **I** and **III** give respectively similar results. Separation of the phosphites is possible in **III** and **IV** only. A two-dimensional chromatogram first with **IV** and then with **II** is illustrated. The relationship between R_F and mol. wt. is discussed including the anomalous behaviour of pyrophosphite. Arsenites and arsenates are treated similarly although arsenites do not give a sharp spot on the chromatogram owing to hydrolysis. E. J. H. BIRCH

467. **Poly- and metaphosphates. III. Application of the technique of paper chromatography to the study of some poly and metaphosphates of which the existence and constitution is in dispute.** J. P. Ebel (*Bull. Soc. Chim. France*, 1953, **20** [11–12], 1089–1095).—A number of phosphates are prepared by known methods and chromatographed on paper either in acid [*iso*propanol (75 ml) - water (25 ml) - trichloroacetic acid (5 g) - 20 per cent. w/v aq. NH_3 (0·3 ml)], or alkaline [*iso*propanol (40 ml) - *iso*butanol (20 ml) - water (3 ml) - 20 per cent. w/v aq. NH_3 (1 ml)] solvents. The "monometaphosphate" described by Beans and Kiehl is shown to be a trimetaphosphate, and that of Pascal to be a mixture of known types, the coagulation of albumin being attributed to Graham's salt. The "dimetaphosphate" of Pascal and Rechid is also shown to be a mixture and that of Travers and Chu to be trimetaphosphate contaminated with pyrophosphate. "Dimetaphosphoric acid" of Travers and Chu prepared by the action of cold water on P_2O_5 contains largely tetrametaphosphate. The "dimetaphosphate" of Laforgue-Kantzer shows a spot for pyrophosphate only. The tetraphosphate of Thilo and Rätz does give rise to a new spot in the polyphosphate region. E. J. H. BIRCH

468. **Poly- and metaphosphates. IV. The acid corresponding to Graham's salt.** J. P. Ebel (*Bull. Soc. Chim. France*, 1953, **20** [11–12], 1096–1099).—The free acid is prepared from Graham's salt by decomposition of the Pb salt with H_2S or by passage through a cation-exchange column of Amberlite IR-100H in the acid state. Chromatography on paper with acid solvent [*iso*propanol (75 ml) - water (25 ml) - trichloroacetic acid (5 g) - 20 per cent. w/v aq. NH_3 (0·3 ml)] and alkaline solvent [*iso*propanol (40 ml) - *iso*butanol (20 ml) - water (39 ml) - 20 per cent. w/v aq. NH_3 (1 ml)] shows that decomposition of the Pb salt leads to considerable depolymerisation, but the resin affords acid in nearly the same state of polymerisation; it is not as stable as the salt. The literature is discussed and a potentiometric titration of the free acids is described. E. J. H. BIRCH

469. **Analysis for industry.** [**Analytical methods for antimony.**] D. Gibbons (*Ind. Chem.*, 1953, **29**, 418–420).—Methods for the determination of antimony are reviewed (52 references). For reference to the first part of this review see *Brit. Abstr. C*, 1953, 462. F. RUMFORD

470. **Analytical reactions of quadrivalent vanadium.** V. I. Kuznetsov and L. S. Kozyreva (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [2], 90–104).—The behaviour of V^{IV} towards 129 inorganic and organic reagents is reviewed. Twenty-eight types of atomic groupings that facilitate reaction with V^{IV} in aq. soln. are mentioned. The reagents are considered in groups according to their effects, *viz.*, pptn. reagents, colour reagents and complex-forming reagents that interfere with other reactions of V^{IV}. G. S. SMITH

471. **Volumetric determination of small quantities of vanadium in aluminium.** G. de Angelis and V. Carunchio (*Ann. Chim., Roma*, 1953, **43** [11], 730–735).—V contained in Al in the proportion of 0·005–0·015 per cent. is determined by pptn. with cupferron in soln. acidified with conc. HCl. The org. substance in the ppt. is destroyed by ignition at 600° C and V_2O_5 is brought into soln. by fusion with $KHSO_4$ and addition of H_2SO_4. V is titrated with Fe^{++} in the presence of oxidised diphenylamine. S. K. LACHOWICZ

472. **A spectrophotometric study of the sodium vanadates and its analytical application.** I. M. Gottlieb (*Dissert. Abstr.*, 1953, **13** [5], 662).—The effect of changes in the pH of aq. solutions on the nature of the complex VO_4' ion is studied by a method of u.v.-absorption spectrophotometry. Aggregation of the ion causes increased transmission of the soln. and this is also effected by increasing the acidity. From results obtained it is suggested that the so-called "metavanadate" ion is in fact a tetravanadate ion. Preliminary studies of the CrO_4'' and PO_4''' systems are reported and also a survey of the spectra of several oxygenated metallic anions in *M* NaOH. Analytical procedures for the determination of traces of V and for determining PO_4''' are presented. Two component analyses of mixtures of Cr and V gave satisfactory results. N. M. WALLER

473. The determination of arsenic as arseni-12-tungstate [12-tungstoarsenate]. H. M. Brazzell (*Dissert. Abstr.*, 1953, **13** [5], 661).—A gravimetric method for the determination of As within the limits of 0·05 to 4·0 mg is described. The AsO_4''' is converted to 12 tungstoarsenate ion by slow acidification with HCl or H_2NO_3 of a soln. containing AsO_4''' and excess of WO_4'' at 60° C. Excess of WO_4'' is removed by pptn. of H_2WO_4 before pptn. of the 12-tungstoarsenate with tetraphenylarsonium chloride or with cinchonine. Cinchonine gives inconsistent results unless the ppt. is ignited to const. wt. at 500° C. Interfering cations include those that form insoluble chlorides or complex chlorides that react with the precipitant; reducing ions also interfere. Anions that react directly with the precipitant must be excluded, *e.g.*, MnO_4', ReO_4', IO_4'; other interfering anions include SiO_3'', PO_4''', SO_4'', citrate, tartrate and acetate. N. M. Waller

474. Separation of tantalum and niobium by solvent extraction. P. C. Stevenson and H. G. Hicks (*Anal. Chem.*, 1953, **25** [10], 1517–1519).—Ta and Nb are separated by di*iso*propyl ketone extraction from aq. solution containing HF and one of the following acids: HCl, HNO_3, H_2SO_4 or $HClO_4$. Ta is far more readily extracted than Nb, the ratio of Ta to Nb extraction coefficients in the solvent being lowest at 91 for HCl - HF medium and highest at 880 for HNO_3 - HF. The H_2SO_4 (6 *M*) - HF (0·4 *M*) system appears to be best for the separation of Ta and Nb from general chemical contamination since only elementary halogen, Se^{VI} and Te^{VI} tend to be extracted. The Nb can be extracted from the aq. soln. by increasing the acidity and the HF concn. A radiochemical application is described, the Ta and Nb content of U samples being determined. G. P. Cook

475. Determination of small amounts of niobium and tantalum using radio-isotope tracer technique. T. F. Boyd and M. Galan (*Anal. Chem.*, 1953, **25** [10], 1568–1571).—Radio-isotope tracer technique was applied to the determination of the efficiency of pptn. steps in the colorimetric determination of Nb and Ta in austenitic steel. The method investigated is a modification of that of of Thanheiser (*Institut Eisenforschung*, 1940, **22**, 260). The results indicate that < 0·01 per cent. is lost with a 2-g sample during the first pptn. stage and in the second stage nearly all the Ta is pptd. and 1 per cent. or less of Nb is lost. Between pH of 2·0 to 9·5 nearly all Ta and Nb is pptd. (> 98 per cent.). Details of the analytical procedure investigated and the radio-isotope technique used are given. G. P. Cook

476. A method of analysis for loosely bound sulphur. A. A. Babineau (*Dissert. Abstr.*, 1953, **13** [5], 660).—The action of aromatic nitro compounds on sodium trithiocarbonate and perthiocarbonate in alkaline soln. results in oxidation of the S to S_2O_3''. The S_2O_3'' is determined by I titration after pH adjustment to 3·5. Nitrobenzene, *p*-nitrobenzoic acid and *m*-nitrobenzenesulphonic acid were used as oxidants, the last named being most satisfactory for the procedure described. N. M. Waller

477. The colorimetric estimation of sulphide ions. H. Koren and W. Gierlinger (*Mikrochim. Acta*, 1953, [3], 220–225).—The molybdenum blue, methylene blue and molybdenum thiocyanate methods for estimating sulphide ions and H_2S are discussed and found unsatisfactory. The use of heavy-metal sulphide soln. is considered, and a modification to a previously described Bi reagent (Treiber, Koren and Gierlinger, *Brit. Abstr. C*, 1953, 111) is given. $Bi(NO_3)_3.5H_2O$ (0·22 g) is dissolved in 25 ml of 3·2 per cent. mannitol soln. Eight ml of pure glycerol are added and then 36 ml of a 2·5 per cent. filtered gum arabic solution. The volume is made up to 100 ml with acetic acid - Na acetate buffer (11 vol. 0·2 *M* Na acetate to 3 vol. 0·2 *M* acetic acid). After allowing it to stand 2 days, the solution is filtered. This reagent will keep for about 3 weeks. J. H. Waton

478. Estimation of sulphide, sulphite and thiosulphate by chloramine-T. A. R. Vasudeva Murthy (*Curr. Sci.*, 1953, **22** [11], 342).—Chloramine-T quantitatively oxidises sulphide and sulphite in acid soln., and can therefore be used for their estimation. An aliquot of soln. containing sulphide, sulphite and thiosulphate is shaken with excess of 0·1 *N* chloramine-T in 2 *N* H_2SO_4 for 5 min. and the excess then determined by adding KI and titrating the liberated I with standard $Na_2S_2O_3$ soln. (let chloramine-T consumed = *x*). In a second aliquot, the sulphide is pptd. as CdS, filtered off, washed and oxidised in acid soln. with chloramine-T (consumption = *y*). The filtrate containing sulphite and thiosulphate is acidified and oxidised with known excess of I soln. and back-titrated with standard $Na_2S_2O_3$ (chloramine-T equiv. of I consumed = *z*). If *a*, *b* and *c* are the moles of sulphide, sulphite and thiosulphate in the aliquot, then—

$8a + 2b + 8c = x$; $8a = y$; and $2b + c = z$,

from which *a*, *b* and *c* can be calculated. Results showing the superior accuracy of the method over the Kurtenacker - Wollack method with standard soln. of I and $Na_2S_2O_3$ only, are given. G. Helms

479. A spectrophotometric method for the determination of sulphate and organic sulphur on the micro- and ultramicro-scale. L. Andersen (*Acta Chem. Scand.*, 1953, **7** [4], 689–692).—An improved method of estimating sulphate pptd. as benzidine sulphate is described. The ppt. is taken up in dil. HCl and analysed spectrophotometrically under 250 mμ light. Accuracy is 1 to 2 per cent. in the range 0·1–1·5 mg of S and rather less in the 1–25-μg range. Details are given together with a discussion of the requirements for successful pptn. of the benzidine sulphate. A. B. Hart

480. Determination of tungsten in complex alloys. R. B. Golubtsova (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [2], 105–109).—The antiseptic Rivanol, 2:5-diamino-7-ethoxyacridine, is recommended as a gravimetric reagent for W. In acid solution a 2 per cent. aq. soln. of the reagent gives with tungstates a cream-yellow ppt. of the composition $(C_{15}H_{13}ON_3)_2.H_2WO_4$. The ppt. coagulates in the cold but more quickly on a bath of boiling water. It can be washed with dil. HCl (1 + 400) and ignited to WO_3. The reagent permits the determination of W in Fe, Ni, Co and Cr-based alloys and in cast irons in presence of large amounts of Ti, Zr, V, etc., Ni-based alloys are dissolved in a mixture of 60 ml of HCl (1 + 1) and 5 ml of conc. HNO_3, other alloys are first gently heated with 80 ml of HCl (1 + 1) and then with 5 ml of conc. HNO_3. The soln. is evaporated to pastiness and then dried. The residue is gently heated for 1 hr. with 50 to 60 ml of HCl (1 + 4), treated with 100 ml of hot water and again heated for 1 hr. The ppt. with paper pulp added is filtered, washed free from Fe with 10 per cent. HCl, placed in the original beaker, mixed with hot aq.

NH_3 soln. (1 + 1), and heated at 100° C for 30 min. The liquid is filtered, the residue is washed with hot dil. aq. NH_3 soln., and the filtrate is made acid to methyl orange with conc. HCl, 3 to 4 drops being added in excess. Five ml of 2 per cent. Rivanol soln. for each 2·5 per cent. of W in a sample weight of 1 g are added. After coagulation the ppt. is filtered, washed with HCl (1 + 400), dried and ignited carefully. G. S. SMITH

481. **Determination of small quantities of tungsten in the presence of molybdenum.** A. de Sousa (*Z. anal. Chem.*, 1953, **140** [3], 170–173).—A quick and simple method of determination of small quantities of W in the presence of a fifty-fold excess of Mo is described. Both metals are pptd. from the soln. as $CaWO_4$ and $CaMoO_4$ and the ppt. is treated with an excess of conc. HCl. $CaMoO_4$ dissolves completely and is filtered from the ppt. of H_2WO_4, which is then dried, ignited and weighed as WO_3. The procedure outlined is entirely satisfactory for determination of W in Mo ore and in steel; the determination takes 3 to 4 hr.

S. K. LACHOWICZ

482. **The micro-determination of chloride ion using an amplification procedure.** R. Belcher and R. Goulden (*Mikrochim. Acta*, 1953, [3], 290–297).—In the titrimetric method for Cl′ the titre can be amplified sixfold by metathesis with an iodate, filtering and completing the estimation iodimetrically. $AgIO_3$, $Hg_2(IO_3)_2$ and $Hg(IO_3)_2$ all give errors $\simeq$ 0 to 2 per cent. for Cl′ in the range 0·7 to 3·5 mg. $Hg(IO_3)_2$ appears superior to the others, but cannot be used in acid solution.

J. H. WATON

483. **Determination of iron in copper alloys by means of dimethylglyoxime in acid medium without taking drillings.** I. I. Kalinichenko (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [2], 110–113).—Addition of 1 per cent. dimethylglyoxime in 5 per cent. NaOH to an Fe solution reduced with $SnCl_2$ gives at a pH > 4·8 a dirty red colour that on addition of HCl to produce a pH $\gtrsim$ 2 turns to a red or pink colour stable for many days. A stable colour is not attained in presence of NH_3 or when the Fe is reduced by SO_2 or N_2H_4. The sensitivity of the colour reaction for Fe is equiv. to that of the thiocyanate reaction. The presence of Mn, Co, Cd, Zn, Pb, Al and alkaline earth elements in amounts $\gtrsim$ 100 times that of the Fe do not interfere. Cu, Bi, Cr and Ni can be tolerated up to 20 times the Fe content. In Cu alloys preliminary separations are necessary. To determine Fe in Cu alloys without drilling, 2 or more drops of HNO_3 (1 + 1) are placed on a cleaned part of the metal. After cessation of the reaction, the soln. is transferred to a tube, the metal surface is washed 2 or 3 times with 2 to 3 drops of water and the washings are added to the contents of the tube together with a drop of H_2SO_4 (1 + 3). The hot soln. is then treated with 0·05 g of dry CdS to ppt. CuS. The soln. is filtered and the filter is washed with hot water. The filtrate is diluted to 10 ml, a suitable vol. is transferred by pipette to a cylinder, 2 drops of conc. HCl, 3 drops of $SnCl_2$ soln. and 3 drops of 20 per cent. Na K tartrate are added, and the solution is boiled. Then 10 drops of dimethylglyoxime soln., 5 drops of 20 per cent. NaOH, and after mixing, 5 to 7 drops of conc. HCl (to dissolve the ppt.) are added. The colour intensity is compared with that of standards. G. S. SMITH

484. **Analytical application of alginic acid as a cation exchanger. [Separation of iron.]** H. Specker and H. Hartkamp (*Z. anal. Chem.*, 1953, **140** [3], 167–170).—Separation of $Fe^{\cdot\cdot\cdot}$ from Mg, Al or UO_2 ions by means of a column filled with alginic acid is described. Owing to the high selectivity of the exchanger towards $Fe^{\cdot\cdot\cdot}$ the separation of Al or U from Fe is easily effected by elution with an acid. Solutions containing excess of acid should be used, as alkali alginates are appreciably soluble in water.

S. K. LACHOWICZ

485. **Determination of tervalent iron by means of tetraborate.** A. K. Babko (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [2], 127–128).—The paper of Schigol *et al.* (*Brit. Abstr. C*, 1953, 157) is strongly and adversely criticised. The experiments were badly arranged and the results were inadequate to give the conclusions stated. G. S. SMITH

486. **The reaction between iron sulphide and sulphur dioxide.** T. Rosenqvist and P. H. Hynne (*Tidsskr. Kemi Bergv.*, 1953, **13** [9], 196–200).—The equilibrium ratio of the reaction $3FeS + 2SO_2 \rightleftharpoons Fe_3O_4 + 5S$ obtained by analysis of gas mixtures in equilibrium with the solid reactants is given by $10^5[p_{S_2}]^{5/2}/[p_{SO_2}]^2 = 2{\cdot}7$ (at 700° C), 7·4 (at 800° C), or 21·8 (at 900° C). A eutectic is present in the FeS - Fe_3O_4 system at 1010° $\pm$ 10° C.

J. H. BURTON

487. **α-Furildioxime as a reagent for gravimetric and colorimetric determination of nickel.** V. M. Peshkova, G. A. Goncharova, E. A. Gribova and I. V. Puzdrenkova (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [2], 114–118).—α-Furildioxime is suitable for microgravimetric determinations of Ni, *e.g.*, for 0·5 mg of Ni in 50 ml of soln. The pH for quant. pptn. is in the range 6·4 to 10·5. In presence of tartrate Fe and Al do not interfere at concn. up to 100 times that of the Ni. Colorimetric determinations can be carried out with chloroform or benzene extracts of the complex from soln. of pH 7·3 to 8·4.

G. S. SMITH

488. ***iso*Nitrosodimedone: a reagent for cobalt.** J. Gillis, J. Hoste and J. Pijck (*Mikrochim. Acta*, 1953, [3], 244–253).—By means of a microcrystallographic technique, *iso*nitrosodimedone is found to be a suitable reagent for detecting Co, yielding a birefringent red crystalline ppt. grouped in the form of rosettes. Most common ions do not interfere, but $Hg^{\cdot\cdot}$ and $Ag^{\cdot}$ need a large excess of reagent, and $Fe^{\cdot\cdot}$, $Fe^{\cdot\cdot\cdot}$ and $UO_2^{\cdot\cdot}$ have to be masked with NaF. The solubility of the Co complex is 4·5 mg per 100 ml, so it is considered to be suitable for the gravimetric micro-estimation of Co. Thermogravimetric analysis indicates that the ppt. is stable up to 260° C. An excess of reagent between 120 and 200 per cent. should be employed, with a min. time of digestion in ice of 2½ hr. The amount of Co should be $\simeq$ 1 mg, and the pH between 5·3 and 6·9. Common ions do not interfere with the estimation, but the proportion of $Cu^{\cdot\cdot}$ must not exceed 10:1, and $Fe^{\cdot\cdot}$, $Fe^{\cdot\cdot\cdot}$ and $Ag^{\cdot}$ must be removed. The following procedure is recommended. Three ml of an aq. soln. containing 0·5 to 3 mg of Co is taken and the pH is adjusted to between 5·3 and 6·9. After warming to 40° to 50° C, a 120 to 200 per cent. excess of reagent (1 per cent. aq. soln.) is added. The temp. is kept constant at 40° to 50° C for 10 min. and then the soln. is put in ice for at least 2½ hr. The ppt. is filtered, washed 3 times with 1 ml of iced H_2O (5 times if other ions present) and dried at 150° to 160° C for 15 min. J. H. WATON

489. **Colorimetric determination of small quantities of cobalt with β-nitroso-α-naphthol.** H. Baron (*Z. anal. Chem.*, 1953, **140** [3], 173–184).—Colorimetric determination of Co in the form of the coloured complex with β-nitroso-α-naphthol dissolved in toluene is described. The complex is first pptd. in water following a standardised procedure and the suspension is shaken with toluene, whereby the complex dissolves and passes entirely into the toluene phase. After washing the toluene soln. with NaOH its extinction is determined in a photometer with a green (530 mμ) filter. The method is adapted for determination of traces of Co in animal fodder and in soil. S. K. Lachowicz

490. **Microtitrations with ethylenediaminetetra-acetic acid. VII. The estimation of palladium.** H. Flaschka (*Mikrochim. Acta*, 1953, [3], 226–228).—A freshly prepared and highly conc. solution of $K_2Ni(CN)_4$ is added in excess to a weakly acid solution of Pd^{++}. After thoroughly stirring it, the solution is made alkaline with dil. aq. NH_3, when the Pd^{++} liberates its equiv. of Ni^{++}. After strong dilution so that the solution is 0·001 *M* to Pd, the Ni^{++} is titrated with the 0·01 *M* complexone solution with murexide as indicator. The max. errors found for Pd are ± 20 μg. J. H. Waton

491. **Estimating oil yield of lean oil shale.** K. E. Stanfield (*Anal. Chem.*, 1953, **25** [10], 1552–1553).—Approximately 3 g of the crushed shales are placed in the bottom of borosilicate test tubes, which are suspended to a depth of 2 in. in an electric muffle furnace with a Transite cover. The furnace is maintained at 600° ± 50° C. After 3 min. water or other condensate is usually observed in the upper parts of the tubes. Oil, if present, is indicated by either odour, condensate or white to brown vapours below the water zone or a dark ring on the tube walls in the refluxing zone. After 5 min. the tubes are removed and the oil yields are estimated from the above observations and by comparison with standards consisting of oil shales prepared in the same manner. G. P. Cook

See also Abstracts 576, 627.

3.—ORGANIC ANALYSIS

492. **Micro-analysis of fluorine-containing organic compounds. II. The determination of fluorine.** R. Belcher, E. F. Caldas, S. J. Clark and A. Macdonald (*Mikrochim. Acta*, 1953, [3], 283–289).—A 3 to 5-mg sample is heated with 30–40 mg of small Na pellets (or K pellets if the material is a fluorocarbon) in a Ni bomb placed in a furnace at 600°–650° C for an hour. The contents of the bomb are made up to 100 ml, filtered and 20-ml aliquots are used. The F′ is estimated with $Th(NO_3)_4$, the usual procedure being modified by adopting a back titration of a comparison solution. To a 20-ml aliquot is added the vol. of 0·05 *N* HCl required to neutralise the soln. to Hoppner's indicator (vol. found for another 20-ml aliquot) together with 2·50 ml in excess. To a comparison tube of distilled H_2O are added 2·50 ml of 0·05 *N* HCl and a vol. of 0·05 *N* NaCl or KCl (according as to whether a Na or K fusion is used in the bomb) equal to that of the 0·05 *N* HCl required to neutralise the 20-ml aliquot. After adding 1 ml of Alizarin Red S indicator soln. to both, the unknown soln. is titrated with 0·001 *M* $Th(NO_3)_4$ until a pronounced pink colour is attained, and the soln. is made up to 50 ml. The same vol. of $Th(NO_3)_4$ is added to the comparison soln., which is titrated back with standard NaF or KF solution (containing 25 μg F′ per ml) to match the first soln. when made up to 50 ml. If N or S is present in the sample, the HCN or H_2S present in the acidified 20-ml aliquot is removed by boiling for 15 to 30 sec. After neutralising with 0·05 *N* NaOH and re-acidifying with 0·05 *N* HCl, the determination is completed as before. J. H. Waton

493. **Application of the dead-stop end-point titration to the Zimmermann micro-estimation of sulphur.** P. Monand (*Bull. Soc. Chim. France*, 1953, **20** [11-12], 1063–1065).—The H_2S formed by the action of K on an S compound and decomposition of the K_2S, is converted into CdS in the Zimmermann method, treated with IO_3' and I′, and the excess of I is titrated with $Na_2S_2O_3$ by a dead-stop method. A diagram of a "magic eye" circuit that sensitively indicates the polarisation of the electrodes is given, and results are tabulated showing results for dithizone, thiourea and dithio-oxamide. E. J. H. Birch

494. **The determination of acetylene in air.** B. J. Purser (*Analyst*, 1953, **78**, 732).—The method previously reported (*Brit. Abstr. C*, 1950, 4) has been improved. The prep. of a reference soln. of acetylene in acetone is described. Aliquots of this are treated with Cu_2Cl_2 and the colour is measured absorptiometrically. A calibration factor can then be calculated if the temp. and pressure at the time of filling the sample tube are known. A. O. Jones

495. **Quantitative determination of ethylene oxide products in aqueous solutions or dispersions.** N. Schönfeldt (*Nature*, 1953, **172**, 820).—The method depends on addition products formed between ferrocyanic acid and oxygen containing org. compounds. $K_4Fe(CN)_6$ is added to the acidified aq. solution or dispersion of the ethylene oxide product. The ppt. is filtered and the $K_4Fe(CN)_6$ remaining in the filtrate is estimated. Accuracy is well within ± 5 per cent. S. M. McLaren

496. **Quantitative determination of 1:2-glycols in mixtures.** E. A. Adelberg (*Anal. Chem.*, 1953, **25** [10], 1553–1554).—The method consists in chromatographing the sample on paper, eluting the identified dihydroxy-compound spots and subsequently determining them colorimetrically after conversion to carbonyl-containing fragments by oxidation with IO_4'. The method as applied to *iso*leucine and valine precursors is described. The solvent system for these is an ether - benzene mixture (70 : 30) made 3 *M* with formic acid and saturated with water. Tartaric acid moves satisfactorily in water-saturated butanol with 3 *M* with formic acid. The compounds are detected by spraying with bromophenol blue and eluted with water. $NaIO_4$ and KI are added and the liberated I is titrated with $Na_2S_2O_3$ solution; 2 : 4-dinitrophenylhydrazine and NaOH are then added, the solution is placed aside for 15 min. and the absorbancy is then measured in a Klett colorimeter with the green (No. 54) filter. The method affords 100 per cent. recovery with max. error of ± 10 per cent. G. P. Cook

497. **Spectrophotometric determination of glycerol as sodium - cupri - glycerol complex.** F. Spagnolo (*Anal. Chem.*, 1953, **25** [10], 1566–1568).—In the preparation of a standard calibration curve, various aliquots from an aq. stock soln. of glycerol were diluted with water to 10 ml, 10 ml of 21 per cent, NaOH soln. and 60 ml of ethanol were added.

followed by addition of 6 ml of $CuCl_2$ soln. (10 per cent. in alcohol) slowly and with vigorous swirling. A portion of this coloured mixture was centrifuged, the clear supernatant soln. was transferred to a cell and the absorbancy was measured at 635 mμ against a reagent blank. In the application to aq. or alcoholic samples, the above treatment is carried out, after neutralisation, if the samples are acid or alkaline. In the analysis of glycerol esters and resinous vehicles, prior treatment is carried out to produce an aq. soln. of glycerol. Ethylene glycol and 1:2-propylene glycol interfere. The error of the method is $\simeq \pm 1$ to 2 per cent. G. P. Cook

498. **A simple volumetric method for routine determination of glycerol.** J. W. B. Erskine, C. R. N. Strouts, G. Walley and W. Lazarus (*Analyst*, 1953, **78**, 630–636).—The method depends upon the oxidation of glycerol at room temp. by $NaIO_4$ to formaldehyde and formic acid. The excess of $NaIO_4$ is destroyed by ethylene glycol and the formic acid is then titrated in an inert atmosphere with 0·1 *N* NaOH to the phenol red end-point. Oxidation is carried out in the dark and CO_2 must be excluded. Some other polyhydric alcohols and sugars interfere but these are not usually present in glycerol-containing products from soap making and fat splitting. Glycols with adjacent hydroxyl groups react but yield formaldehyde only.

A. O. Jones

499. **Use of dimedone for spot detection of aldehydes.** L. M. Kulberg and I. S. Mustafin (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [2], 122–126).—Use is made of the acidic character of the enol compound formed in the dimedone reaction with aldehydes. The test is carried out on filter-paper, the acid detector being Feigl's Ag_2CrO_4 - NH_3 reagent (*Ind. Eng. Chem., Anal Ed.*, 1942, **14**, 519). G. S. Smith

500. **The determination of *iso*butyraldehyde in *n*-butyraldehyde.** G. R. Primavesi (*Analyst*, 1953, **78**, 647–651).—Sodium borohydride reduces *iso*-butyraldehyde and *n*-butyraldehyde to the corresponding alcohols. The *iso*butanol is then determined absorptiometrically by means of a modification of the Komarowsky reaction for higher alcohols with conc. H_2SO_4 and salicylaldehyde. By an examination of the effects of the variables in this reaction the reproducibility of the method has been improved. A. O. Jones

501. **A qualitative test for monosaccharides.** R. M. Love (*Analyst*, 1953, **78**, 732–733).—The pink colour formed when glucose is heated for a few min. with dil. H_2SO_4 is due to condensation of 5-hydroxymethylfurfural with the groups on C atoms 2 and 3 in the sugar mol. The colour is intensified if 5-hydroxymethylfurfural is added to the glucose before heating. After proving the absence of pentoses, polysaccharides and ketohexoses, the 3 aldoses can be distinguished by this test. To 1 ml of 5-hydroxymethylfurfural in a test tube is added 1 ml of the sugar soln. (0·2 to 4 mg per ml). The tube is placed in iced water and 2 ml of H_2SO_4 are added from a burette with constant swirling that is continued until the tube is cold. A further 4 ml of acid are then added with swirling. A pink colour indicates mannose. If the liquid is colourless the tube is placed in boiling water for $\simeq$ 3 min. A pink colour then indicates glucose and a brown colour galactose. Reactions with other sugars are described, but the test is most useful for hexoses in low concn.; it is more delicate than the osazone test. A. O. Jones

502. **New specific reagent for keto-sugars.** R. Johanson (*Nature*, 1953, **172**, 956–957).—The use of anthrone as a specific spot-test reagent for the identification of ketoses is described. The reagent (300 mg of anthrone warmed with 10 ml of glacial acetic acid, 20 ml of ethanol, 3 ml of H_3PO_4 and 1 ml of water) is sprayed on a paper treated with the test solution. On heating at 108° C for 5–6 min. mono-, di- and tri-polysaccharide-containing ketohexoses develop a bright yellow spot in white light. Keto-xylose and triketo-heptoses tested gave a purple and orange colour, respectively. The variation of depth of colour allows some quant. estimation. The reaction is sensitive, 5 μg of mono-ketose being sufficient for identification.

N. M. Waller

503. **A new colorimetric reagent for carbohydrates.** E. Lunt and D. Sutcliffe (*Biochem. J.*, 1953, **55** [1], 122–126).—The use of a new colorimetric reagent, resorcinol-4:6-disulphonic acid (**I**), for the determination of hexoses and their polysaccharides is described. It is based on the spectrophotometric determination of the optical density at 490 mμ of the golden-orange colour produced on addition of **I** (as Ca salt) and conc. H_2SO_4 to a solution of the carbohydrate after calibration of the reagent with the appropriate hexose standard. Some factors affecting the accuracy of the method have been investigated. The reagent has been used chiefly for the determination of glucose, fructose and their polysaccharides. From 10 to 250 μg of glucose in 5 ml may be determined with errors of less than 1 per cent. Inulin and starch give 100 per cent. and dextran 95 per cent. of the colour intensity given by their constituent monosaccharides. Other reagents that have been tested for the estimation of carbohydrates are briefly discussed.

P. Chaplen

504. **Photometric determination of aldoses in presence of ketoses.** F. Stitt, S. Friedlander, H. J. Lewis and F. E. Young (*Abstr. 124th Meeting Amer. Chem. Soc.*, Sept., 1953, p.12D; *Sugar Ind. Abstr.*, 1953, **15** [10], 727).—Glucose is accurately determined in 0·05–1·0 per cent. admixture with fructose by its selective oxidisability by means of buffered aq. $HClO_2$. ClO_2 is formed and measured with a spectrophotometer or colorimeter, the difference between the results with a reference sample and the test sample being a measure of the glucose content. The method can be extended (with some loss of accuracy with increasing glucose content) to the analysis of any glucose - fructose mixture, and probably also to aldose - ketose mixtures in general. For fructose containing $<$ 1 per cent. of glucose, the concn. of fructose in the test soln. should be 100 mg per ml. P. S. Arup

505. **The quantitative analysis of mixtures of mono- and di-saccharides.** W. M. Corbett (*Chem. & Ind.*, 1953, [48], 1285).—The total mono- and di-saccharides in the presence of each other are determined (max. error $\pm$ 5 per cent.) from the reducing power of the solutions before and after separation on a column of equal parts of acid-washed charcoal and Celite 535. The reducing power is measured by the method of Hagedorn and Jensen modified by addition of 1 ml of 10 per cent. KI soln. to the ferricyanide solution after this has been heated with the sugar solution, then addition of 3 ml of a solution containing 10 g of $ZnSO_4$ and 50 g of NaCl per 200 ml of H_2O and, finally, addition of 2 ml of 3 per cent. aq. acetic acid; 0·002 *N* $Na_2S_2O_3$

is used to titrate the liberated iodine (blank, approx. 5 ml). With a column 3·0 cm × 0·8 cm (diam.) the max. amount of carbohydrate material that can be efficiently separated is 0·4 mg, and from this amount the whole of the monosaccharide is eluted in the first 10 ml of eluate whilst no disaccharide is eluted in 15–20 ml of eluate unless its concn. is high. D. BAILEY

506. **The identification and determination of the lower straight-chain fatty acids by paper partition chromatography.** R. E. B. Duncan and J. W. Porteous (*Analyst*, 1953, **78**, 641–646).—An improved method based upon that of Reid *et al.* (*Brit. Abstr. C*, 1952, 100) for detecting and determining the C_2–C_6 straight-chain fatty acids is described. Paper chromatograms in *n*-butanol - NH_3 mixtures are sprayed with a methyl red - bromothymol blue mixed indicator in formaldehyde soln. (40 per cent.). Each of the possible variable factors in the procedure was exhaustively investigated and the optimum conditions were established for obtaining reproducible chromatograms for the range 5 to 80 μg of each acid. The method has been applied to investigation of volatile fatty acid production in laboratory grass-water fermentation mixtures and in field silage and also to examination of the metabolic products of *Actinomyces israeli*.
A. O. JONES

507. **Determination of formic and glycollic acids formed during the periodic [acid] oxidation of organic compounds.** P. Fleury, R. Perlès and L. Le Dizet (*Ann. Pharm. Franç.*, 1953, **11** [9–10], 581–588).—The solution resulting from a HIO_4 oxidation is treated with powdered cryst. $Ba(OH)_2$ to remove HIO_3 and HIO_4, and excess of Ba is then pptd. from an aliquot with H_2SO_4. The aliquot is then extracted with ether for 2 hr. and the formic acid in the ether layer is extracted with 0·25 *M* Na_2CO_3 soln. (*N* NaOH may be used if glycollic acid is not present). If formaldehyde is present in the original oxidised solution a saturated solution of $(NH_4)_2SO_4$ is added to prevent its extraction by the ether, but the formic acid then requires extraction for 4 hr. The alkaline solution is then warmed to remove residual ether, acidified with *N* H_2SO_4 and treated with $HgCl_2$ - Na acetate solution and a little $BaSO_4$ suspension. After 20 min. in a bath of boiling water, the suspension containing Hg_2Cl_2 is cooled and centrifuged. The centrifuge is washed and treated with excess of KIO_3 - H_2SO_4 and then with KI; the excess of I is titrated with 0·01 *N* $Na_2S_2O_3$. To estimate the glycollic acid, the solution is prepared and extracted as for formic acid except that the ether extraction requires 8 hr.; alkaline extraction is with *N* NaOH. The alkaline solution is acidified and the glycollic acid is determined colorimetrically with chromotropic acid at 520 mμ (*cf.* Fleury *et al. Brit. Abstr. C*, 1951, 369). The error is ± 3 per cent. for the formic and somewhat larger for the glycollic acid. E. J. H. BIRCH

508. **Paper microchromatography of non-volatile water-soluble aliphatic acids. III. New pairs of solvent phases (alkaline and acid) for two dimensional chromatography.** R.-I. Cheftel, R. Munier and M. Macheboeuf (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1085–1089).—Mixtures of aliphatic acids are chromatographed on paper (Durieux 122) first in alkaline and then in acid soln. This order avoids the lack of sharpness caused by development after an alkaline solvent is used. The alkaline solvent used is 2-ethoxyethanol - 20 per cent. w/v aq. NH_3 - water (80 : 5 : 15) and 2-methoxyethanol - cineole (1 : 1) with formic acid or propanol - eucalyptol (1 : 1) with formic acid is used as acid phase. Alternatively, ethanol - 20 per cent. w/v aq. NH_3 - water (80 : 5 : 15) is used as the alkaline phase with 2-ethoxy- or 2-methoxy-ethanol - cineole (1 : 1) with formic acid as acid phase. The acid phases are all made up with 20 ml of formic acid per 100 ml of solvent and with sufficient water to saturate the phase. R_F for each of the solvent phases are quoted for malonic, lactic, succinic, fumaric, tartaric, oxalic, malic, citric, maleic, glycollic, aconitic, α-oxoglutaric, adipic and sebacic acids. The spots are developed by spraying, after drying and evaporation of the formic acid, with 0·1 per cent. bromocresol green in ethanol.
E. J. H. BIRCH

509. **Paper microchromatography of non-volatile water-soluble aliphatic acids. IV. Development technique for dicarboxylic acids.** R.-I. Cheftel, R. Munier and M. Macheboeuf (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1091–1093).—The satisfactory separation of linear dicarboxylic acids by paper chromatography requires an alkaline solvent containing at least 15 per cent. water, under which conditions development of the spots is usually not sharp. Development is sharp by spraying the dried chromatogram with an 0·1 per cent. soln. of bromocresol green - ethanol at its change-point colour, and then with a saturated neutral soln. of Pb acetate in aq. ethanol. The spots appear yellow-green on a violet ground. R_F are quoted for oxalic, malonic, succinic, glutaric, adipic, pimelic, suberic, azelaic and sebacic acids by use of ethanol or 2-ethoxyethanol (80 per cent.) with 20 per cent. w/v aq. NH_3 (5 per cent.) and water (15 per cent.).
E. J. H. BIRCH

510. **The spectrophotometric determination of long-chain fatty acids containing ketonic groups with particular reference to licanic acid.** A. Mendelowitz and J. P. Riley (*Analyst*, 1953, **78**, 704–709).—The spectrophotometric method of Lappin *et al.* (*Brit. Abstr. C*, 1951, 289) based on the colour reaction of ketone groups with alkaline 2 : 4-dinitrophenylhydrazine was examined critically and finally modified. The improved method was applied to the determination of licanic acid in presence of other unsaturated fatty acids. A. O. JONES

511. **Investigation of citric and *iso*citric acids in presence of each other by paper microchromatography.** R.-I. Cheftel, R. Munier and M. Macheboeuf (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1095–1099).—Citric and *iso*citric acids give a similar R_F in a number of different solvents, but if the solutions are evaporated to dryness and heated to 100° C *in vacuo* (water-pump), the *iso*citric acid only is converted into its lactone which has a different R_F. The conversion can be completed by elution of the spots and reheating *in vacuo*. The solvent phase used in the experiment described is ethyl acetate (40 ml), methyl benzoate (10 ml), 90 per cent. aq. formic acid (5 ml) and sufficient water to saturate the phase. E. J. H. BIRCH

512. **Further research on the chromatographic determination of keto-acids.** D. Cavallini and N. Frontali (*Ric. Sci.*, 1953, **23** [4], 605–608).—The method for the chromatographic estimation of keto-acids suggested by the authors (*Nature*, 1949, **164**, 792) is once more subjected to examination. The results confirm original findings.
P. P. BIRNBAUM

513. **Determination of aldonic acids in alkaline media.** J. V. Karabinos, A. T. Ballun and R. L. McBeth (*Anal. Chem.*, 1953, **25** [10], 1563).—Approximately 1 millimole of aldonic lactones or salts is placed in an Erlenmeyer flask and dissolved in 10 ml of 3 per cent. NaOH. The mixture is titrated slowly with 0·106 *M* $CaCl_2$ soln. until turbid. The end-point is attained when the turbidity persists and the soln. becomes more opalescent. The mole ratios of calcium to aldonate are given for several aldonic acids. G. P. COOK

514. **The analysis of acid chlorides.** C. C. T. Chinnick and P. A. Lincoln (*Analyst*, 1953, **78**, 675–678).—A method is described for the analysis of organic acid chlorides containing, as impurities, free organic acid, acid anhydride and HCl. The sample is first esterified with butanol in presence of pyridine and, after dilution with ethanol, is titrated with *N* KOH, a pH meter being used. If free acid or anhydride is present a point of inflection on the titration curve at $\simeq$ pH 6 corresponds to the neutralisation of the HCl and a second at $\simeq$ pH 10 to that of the organic acid. The ester is then hydrolysed with excess of *N* KOH and the excess of alkali is titrated with *N* HCl. Formulae for calculation of results are given. An alternative but less convenient titration procedure with a double indicator is described for use in the absence of a pH meter. A. O. JONES

515. **Determination of esters of phenylcarbamic acid.** Yu. A. Baskakov and N. N. Melnikov (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [2], 119–121).—Hydrolysis of methyl and ethyl esters of phenylcarbamic acid by means of aq. alcoholic KOH and of *iso*propyl esters of phenylcarbamic acid and 3-chlorophenylcarbamic acid by boiling with conc. HCl yields aniline or the corresponding amine, which can be titrated with $NaNO_2$ soln. G. S. SMITH

516. **Constant current coulometric method in the argentometric titration of thiourea.** M. Nakanishi and H. Kobayashi (*Bull. Chem. Soc. Japan*, 1953, **26** [7], 394–396).—The sample of thiourea is treated with excess of ammoniacal AgBr soln., when the thiourea liberates its equiv. of Br'. After acidification with H_2SO_4, the Br' is titrated with $Ag^{\cdot}$ produced by constant-current electrolysis, the reaction being followed potentiometrically. With $\simeq$ 4-mg amounts of thiourea the mean error is — 0·008 mg with a standard deviation of 0·01 mg. The method is capable of estimating smaller amounts of thiourea ($\simeq$ 0·01 mg). The simultaneous determination of thiourea and NH_4CNS in a mixture is also possible by this method when the material is dissolved in 80 to 90 per cent. acetone. J. H. WATON

517. **Titration of N-carboxy-alpha-amino acid anhydrides in non-aqueous solvents.** A. Berger, M. Sela and E. Katchalski (*Anal. Chem.*, 1953, **25** [10], 1554–1555).—A sample of 0·1 to 0·5 millimole of α-carboxyamino-acid anhydride is dissolved in 10 ml of solvent, thymol blue indicator is added and the soln. is titrated with 0·1 *N* Na methoxide to the deep blue end-point. The appearance of a red colour indicates contamination by free acids. The solvents used are methanol, benzene - methanol, acetone, ether, $CHCl_3$, ethyl acetate, acetophenone, dimethylformamide, pyridine, aniline and butylamine. Equiv. wt. data of several α-carboxyamino-acid anhydrides in dioxan and other solvents are also given. G. P. COOK

518. **The colorimetric determination of indene.** W. Roman and M. Smith (*Analyst*, 1953, **78**, 679–680).—Indene can be determined by means of the yellow colour of the fulvene produced with benzaldehyde in alkaline solution. *cyclo*Pentadiene and most other hydrocarbons with a similarly reactive methylene group can be separated from indene by distillation. After reaction under controlled conditions, the optical density is measured absorptiometrically. Readings with standard indene solutions are reported and from these a standardisation graph can be constructed. A. O. JONES

519. **Determination of phenols by the *p*-nitrosodimethylaniline method. [Use of *p*-aminodimethylaniline oxalate and potassium ferricyanide.]** I. Nusbaum (*Sewage Ind. Wastes*, 1953, **25**, 311–313).—A modification of the procedure of Hill *et al.* (*Brit. Abstr. C*, 1953, 276) for the determination of phenol by the indophenol method is described. The reduction of dimethyl-*p*-nitrosoaniline just before its use in the reaction is obviated by using as reagent a 0·1 per cent. soln. of *p*-aminodimethylaniline oxalate in 1 per cent. H_2SO_4 (which is stable for an indefinite period). To a sample containing 5 to 40 μg of phenols a borax buffer is added, then 8 per cent. $K_3Fe(CN)_6$ (to destroy sulphides, which are a major source of interference) and the reagent. The final pH of the mixture should be $\simeq$ 8·3 to 8·7. The colour is allowed to develop for $\not<$ 15 min. and then extracted (*cf.* Hill, *loc. cit.*) with *iso*pentanol, CCl_4, or $CHCl_3$. When interference from aromatic amines is suspected the sample is agitated for 3 to 5 min. with $\simeq$ 5 g of a strongly acidic ion-exchange resin, *e.g.*, Dowex-50-X-S, and then filtered before continuation of the procedure. J. M. JACOBS

520. **Determination of hydrazines, aldehydes and their sulphonic acids using orange G and chromasone red as indicators.** R. Meyer (*Z. anal. Chem.*, 1953, **140** [3], 184–185).—The method described involves titration of hydrazines, aldehydes or their sulphonic acid deriv. dissolved in water or in glacial acetic acid with a standardised solution of phenylhydrazine hydrochloride or benzaldehydesulphonic acid in the presence of Na acetate. The dyes—orange G and chromasone red—are used as external indicators on a piece of filter-paper. S. K. LACHOWICZ

521. **Optical crystallographic properties of organic compounds. IV. 2-Phenyl-1:2:3:2H-triazol-4-ylcarbinol and its oxidation products.** R. N. Castle (*Mikrochim. Acta*, 1953, [3], 196–199).—The optical properties of 2-phenyl-1:2:3:2H-triazol-4-ylcarbinol and of the aldehyde and carboxylic acid obtained on oxidation are examined, the behaviour of the compounds on the micro hot stage is followed, and the micro m.p. are recorded. The optical properties differ sufficiently to permit their use for the positive identification of the compounds. 2-Phenyl-1 : 2 : 3 : 2H-triazol-4-ylcarboxyaldehyde shows a very large positive birefringence ($> 35{\cdot}6 \times 10^{-2}$). No clear-cut trends in optical properties are found in this oxidation series. J. H. WATON

522. **Photometric determination of thiophen in benzene.** E. Giovannini (*Ann. Chim., Roma*, 1953, **43** [11], 736–743).—A photometric determination of thiophen in benzene based on the formation of a coloured alloxan - thiophen complex is described. Owing to the low stability of the complex the determination has to be made after a definite standardised time from the start of the reaction.

The sensitivity of the method is limited to concn. > 0·1 mg per ml. S. K. LACHOWICZ

523. **Chromatographic separation and identification of photographic developers.** J. H. Pannell and J. E. LuValle (*Anal. Chem.*, 1953, **25** [10], 1566).—Standard chromatographic equipment is used in the method and the solvent is a mixture of butanol, acetic acid and water in proportions 4:1:5 by vol. The spray reagents used are 2 per cent. ammoniacal $AgNO_3$ and 5 per cent. molybdophosphoric acid. Limit of detection is 1 μg, best results being obtained at the 10-μg level. R_F values for several developers are listed. G. P. COOK

524. **Coulometric titration of *p*-methylaminophenol sulphate and hydroquinone by cerium (IV).** N. H. Furman and R. N. Adams (*Anal. Chem.*, 1953, **25** [10], 1564–1565).—Approximately 20 ml of saturated $Ce_2(SO_4)_3$ in 2 *M* H_2SO_4 is placed in the titration cell and N is bubbled through for 2 to 3 min. The soln. potential is then raised to the pre-set indicator potential by generating Ce····. The unknown sample of hydroquinone or *p*-methylaminophenol sulphate (**I**) is added after adjustment, the clock is reset to zero, and the Ce···· are generated until zero current is indicated in the galvanometer circuit. The indicator voltage chosen for both titrations is 1·0 V *vs.* hydrogen. The indicator electrode used is 1 sq. cm. of Pt - Ir foil and the reference electrode is a saturated Pb amalgam - $PbSO_4$ - 2 *M* H_2SO_4 electrode. The generator electrode is 2 sq. cm of Pt - Ir foil and the cathode a small platinum wire. The errors for the hydroquinone ranges of 203 to 288, 101 to 134, 63 to 88, 12·5, and 1·28 to 2·33 μg are of the order $\pm$ 0·5, $\pm$ 1·0, $\pm$ 1·0, $-$ 3·2 and $+$ 2·5 per cent., respectively. For **I** the error is $\simeq$ $+$ 1·9 per cent. for 110 μg, $+$ 1·0 per cent. for 68 μg, $+$ 2·0 per cent. for 27 and 38 μg, $+$ 4·7 per cent. for 10 μg, $-$ 2·1 per cent. for 9 μg and $+$ 10 per cent. for 4 μg. G. P. COOK

525. **Determination of the fatty matter in wool by extraction with diethyl ether.** F. F. Elsworth and J. Barritt (*J. Text. Inst.*, 1953, **44** [11], P 754–759).—The importance of factors such as Soxhlet capacity, sample weight, number and rate of siphonings in the determination of fatty matter in wool has been experimentally determined. It is only necessary to specify the max. sample weight for a given barrel size. The min. ratio of 10:1 for solvent to wool is suggested. Removal of fatty matter from dry-combed top by this process is slow and incomplete and the number of siphonings is therefore important in attaining reproducibility. E. S. LANE

526. **The analysis of rosin size.** D. E. Davies and K. Linke (*Analyst*, 1953, **78**, 670–672).—Methods are suggested for the determination of all the constituents of rosin size. They include methods for determination of free alkali and for free rosin acid in presence of free alkali carbonate, a condition rendered possible by the reversibility of the reaction. Results indicate that the proportions of free acid and alkali present may be large. A. O. JONES

527. **Determining rubber hydrocarbon in rubber-bearing plants.** J. W. Meeks, R. V. Crook, C. E. Pardo, jun., and F. E. Clark (*Anal. Chem.*, 1953, **25** [10], 1535–1538).—A modification of the conventional method is described, the essential features being plant material separation, benzene extraction by means of a mechanical shaker and hydrocarbon determination by bromination of the benzene rubber soln. Comminution is carried out by crushing and sheeting through corrugated and smooth rolls and benzene extraction by mechanical shaking with pebbles and trichloroacetic acid (1 per cent. in benzene). Complete analysis takes less than a day and accuracy and precision are higher than by the triple-solvent method. G. P. COOK

528. **The ash of neoprene compounds with special reference to zinc oxide.** H. J. Stern and D. Hinson (*India Rubber J.*, 1953, **125** [23], 62–63).—A wet oxidation method for the estimation of ZnO in neoprene compositions is given. One gram of sample is decomposed in a Kjeldahl flask with 5 to 10 ml of HNO_3 until the soln. clears. The soln. is then heated with 2 ml of H_2SO_4, with dropwise addition of a little HNO_3, until SO_3 is evolved. The residue is diluted with water, transferred to an evaporating basin and evaporated nearly to dryness. The moist residue is taken up with 50 ml water, filtered if necessary, and Pb is removed, if present, with H_2S. The resulting soln. is treated with 5 ml of 5 *N* NH_4Cl and made up to 100 ml. Fe and Al_2O_3 are removed by boiling, making alkaline and filtering. The filtrate is acidified with formic acid and H_2S is passed in at 80° C to complete pptn. The ppt. is washed with hot water, transferred to a weighed silica crucible, moistened with 2 ml 5 *N* H_2SO_4, dried at 110° C, then charred and ignited to ZnO to constant wt. J. L. PROSSER

529. **Chemical analysis of GR-S [synthetic rubber] by complete solution procedures. Gross constituents in GR-S containing soap.** F. J. Linnig, J. M. Peterson, D. M. Edwards and W. L. Acherman (*Anal. Chem.*, 1953, **25** [10], 1511–1515).—The organic acid and soap content of the synthetic rubber are determined by titration of aliquots of a toluene - ethanol (5 + 1) soln. of the sample, the bound styrene by measurement of the refractive indices of the polymer purified by pptn. from the titrated soap aliquot and the stabiliser content by spectrophotometric measurement on a third aliquot. For the stabiliser determination, the rubber solution is diluted with methyl*cyclo*hexane and the λ used are 309 mμ for PBNA (phenyl-2-naphthylamine) stabiliser and 288 mμ for BLE (a reaction product of acetone and diphenylamine) and Stalite (a heptylated diphenylamine) stabilisers. In the determination of organic acid and soap, aliquots are titrated to *m*-cresol purple with standard acid or alkali. The bound styrene is pptd. from a propanol - water mixture and then extracted into anhydrous ethanol - toluene azeotrope; the refractive index is then measured. Accuracy for the soap and acid titrations is better than 1 per cent. (relative), and within 1 per cent. for the stabiliser determination. The method is only applicable to uncompounded GR-S coagulated with salt and acid containing no excess of mineral acid. G. P. COOK

530. **Chemical analysis of GR-S [synthetic rubber] by complete solution procedures. Titration of mineral and organic acids in toluene - ethanol solution.** F. J. Linnig and A. Schneider (*Anal. Chem.*, 1953, **25** [10], 1515–1517).—Both types of acid in the sample are determined by titration to two different end-points by means of the same indicator. A 2-g sample is dissolved in 140 ml of toluene and 30 ml of 95 per cent. ethanol are added with swirling, followed by addition of *m*-cresol purple indicator and titration with 0·1 *N* NaOH (alcoholic). A pink

colour indicates mineral acid, which is determined by titration to the yellow end-point; the organic acid is determined by continuation of the titration to the purple end-point. The mineral acid is determined as per cent. H_2SO_4 and the organic acids as stearic or rosin acid. The method is accurate within the limits of precision of measurement. G. P. Cook

See also Abstract 443.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

531. **Determination of trypsin inhibitor in blood plasma.** S. F. McCann and M. Laskowski (*J. Biol. Chem.*, 1953, **204** [1], 147–152).—In an attempt to apply the method of Kunitz (*J. Gen. Physiol.*, 1947, **30**, 291) to the determination of trypsin inhibitor in blood-plasma, the salt concn. of the diluent was found to exert a significant influence. When physiological saline was used as a diluent a stoicheiometric non-dissociable complex was formed between trypsin and blood plasma trypsin inhibitor. When the concn. of salt was below 0·03 *M* the straight proportionality between the amount of inhibitor and amount of trypsin inhibited no longer existed; in such media the relationship was represented by complicated S-shaped curves.
P. N. Campbell

532. **Distillation of microquantities of iodine. Application to determination of protein-bound iodine in bovine blood serum.** G. H. Ellis and G. D. Duncan (*Anal. Chem.*, 1953, **25** [10], 1558–1559).—Protein-bound I is separated from serum by $ZnSO_4$ pptn., the iodine being finally obtained in 70 per cent. H_2SO_4. Digestion with conc. H_2SO_4 and chromic acid at 200°–220° C then oxidises organic matter and converts I to iodate. The soln. is cooled below 100° C, water is added and the soln. is heated to boiling. H_3AsO_3 and H_3PO_3 are drawn into the distillation flask and the I is distilled over into NaOH containing Na_3AsO_3 by means of a strictly controlled air flow. The I is then determined colorimetrically after reaction with H_3AsO_3 and ceric ammonium sulphate. Recoveries from serum containing added I are between 92·5 and 100 per cent. with a mean of 96 per cent. A diagram of the distillation apparatus used is also given.
G. P. Cook

533. **Flame photometric ultramicro-analysis of serum Na, K and Ca.** R. Herrmann (*Z. ges. exp. Med.*, 1953, **122**, 84–89).—A previously described flame photometric method (*Brit. Abstr. C*, 1952, 343) is elaborated to allow determination of Na, K and Ca in 0·005 ml of serum. Serum is diluted with 100 parts (instead of 10) of a soln. made of 996 ml H_2O and 4 ml of soln. containing 26·617 g of Li_2CO_3 plus HCl in 1000 ml of H_2O. Photometric analysis of this more dilute soln. required higher lead resistance or increased amplification of the photo-electron amplifier. Metal-interference filters are used in combination with colour filters. Working details are given. Capillary tubes for measuring 0·005 ml serum were calibrated relative to the pipette containing approx. 0·495 of the diluent, and accuracy was attained by flame photometric analysis in comparison with serum diluted with macro-pipettes. The mean percentage error for analysis of the diluted serum was for Na 0·3, K 2·0 and Ca 1, and the mean percentage error including dilution of serum was for Na 0·7, K 2·3 and Ca 2·2. P. F. Meyer

534. **Reduction of cupric - EDTA complex by sugars.** M. Reiner and J. Preiss (*Baskerville Chem. J.*, 1953, **4**, 15–17; *Sug. Ind. Abstr.*, 1953, **15**, 805).—The reduction by sugars of the Cu complex of ethylenediaminetetra-acetic acid (**I**) is compared with that of the Cu - citrate (Benedict) complex reagent. A suitable reagent (with reaction time for reducing sugars of 5 to 10 min. on a bath of boiling water) is prepared by adding a soln. (optimum) of the di- or tetra-sodium salt of **I** to aq. $CuSO_4.5H_2O$ and making the mixture alkaline with NaOH (with which the optimum working pH of 12 is reached) or with Na_2CO_3 (for max. sensitivity). Non-reducing carbohydrates do not react, but $CHCl_3$ reduces the reagent **I** when this is made with NaOH but not when made with Na_2CO_3. Reduction occurs only at pH $>$ 9. In tests with artificial diabetic urine (containing 0·05 to 5·0 per cent. of glucose), the EDTA reagent is slightly less sensitive than Benedict's reagent. P. S. Arup

535. **Estimation of nicotinic acid in urine.** D. K. Chaudhuri (*Ann. Biochem. Exp. Med.*, 1953, **12**, 119–122).—The method of Chaudhuri (*Indian J. Med. Res.*, 1951, **39**, 401) for this determination, in which the column is developed with *p*-aminobenzoic acid, fails with samples of urine containing much sugar, as the extracts are appreciably coloured and extinction coefficients are determinable only with difficulty. The method is modified by incorporation of the *iso*butanol extraction method of Lang and Kodicek (*Biochem. J.*, 1943, **37**, 530) and oxidation with $KMnO_4$ before addition of the reagent. Replication on a single sample gives precision of — 10 to + 18 per cent. for nicotinic acid or amide; recovery for 50-mg amounts is 98 to 104 per cent.
L. G. L. Unstead-Joss

536. **Urinary excretion of acid phosphates.** O. Daniel, P. R. N. Kind and E. J. King (*Brit. Med. J.*, 1954, **i**, 19).—Urinary acid phosphatase was determined by dialysing the 20-ml sample in parchment shells against cold running tap water, the vol. being measured after dialysis and any change being accounted for in the final calculation. The dialysed urine was diluted, usually four times, and the enzyme content was estimated by a slightly modified King - Armstrong method. The dialysed urine (0·5 ml) was added to a substrate buffer mixture consisting of 1 ml of 0·02 *M* disodium phenylphosphate, 2 ml of sodium citrate - citric acid buffer, pH 4·9, and 1 ml of 5 per cent. neutral formaldehyde soln. and incubated at 37° C. After 1 hr., 1·5 ml of dil. Folin - Ciocalteu reagent (1 + 3) and 3 ml of 15 per cent. Na_2CO_3 soln. were added and after further incubation for 15 min. at 37° C the colour was measured in a photo-electric colorimeter with an Ilford 608 red light filter. N. E.

537. **Determination of fluorine in bones and teeth.** H.-D. Cremer and W. Voelker (*Biochem. Z.*, 1953, **324** [2], 89–92).—A method is described for determining between 5 and 50 μg of F in bones and teeth. The F is distilled off by treatment of the material with $HClO_4$ and is determined colorimetrically in the distillate as H_2SiF_6 by measurement of the diminution of colour of the ferric salicylate complex. For quantities of F up to 5 μg the error is ± 4 per cent. and for large quantities ($>$ 15 μg) ± 2 per cent. A. J. Mee

538. **Spectrochemical determination of magnesium in teeth.** H. J. Eichhoff, G. Dittmann and H.-D. Cremer (*Biochem. Z.*, 1953, **324** [1], 32–35).—The method entails the use of electrodes of pure electrolytic Cu, and Cr as a comparison element, the lines Mg^{II} 2795·5 A to Cr^{II} 2835·6 A and Mg^{II} 2802·7 A to Cr^{II} 2835·6 A being chosen. About 10 mg of the tooth material was dissolved in 0·3 ml HCl (≃ 25 per cent.), 0·5 ml of a soln. of $CrCl_3$ (1·2 g Cr per litre) was added, and 0·01 ml of the mixture was placed on three purified copper electrodes and dried. The electrodes were then used in conjunction with a quartz spectrograph. The method gives an accuracy of ± 7 per cent. if one pair of lines is used for comparison and ± 5 per cent. if two pairs are used.
A. J. Mee

539. **Microchemical determination of iodine content of water in the eye-socket and the possibility of its extended use with the aid of radio-iodine.** H. Spitzy and H. Skrube (*Biochem. Z.*, 1953, **324** [1], 60–65).—A sample (0·1–0·3 g) of the water is mixed with 1 ml of an aq. soln. containing 1 μg of I (as KI), 1 drop of 13 per cent. K_2CO_3 soln. is added per 0·1 g of test liquid and the mixture is evaporated to dryness and heated to 400° C. It is then dissolved in water and 200 ml of a soln. of acetic acid and Na acetate and 2 drops of Br water are added. After buffering with 200 ml of 20 per cent. Na acetate, the excess of Br is removed by the addition of 1 drop of formic acid soln. and the soln. is warmed for 1 min. After addition of 5 drops of 0·5 per cent. KI soln. the I is determined by titration with 0·005 *N* $Na_2S_2O_3$. The method is satisfactory for concentrations of I up to 0·09 μg in 0·1 g of H_2O. To determine minute traces of I the chemical method can be combined with the isotope technique.
A. J. Mee

540. **Paper chromatography of the carbohydrate constituents of cerebrosides from a Gaucher spleen.** J. Montreuil, P. Boulanger and E. Houcke (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1125–1127).—A normal spleen, a spleen from a case of Gaucher's disease and some normal human brain are dehydrated for 4 months at 4° C under acetone and the cerebrosides are extracted from the powdered tissue by established methods and then hydrolysed with 2 *N* HCl at 100° C for 45 min. After removal of the lipoids with ether, the solutions are passed through columns of Permutit 50 and De-acidite 200, concentrated and then chromatographed on paper by means of pyridine - ethyl acetate - water (1 : 2 : 2) as the mobile phase; the spots are developed with aniline oxalate reagent. It is shown that the proportion of glucose to galactose is greatly increased in the cerebrosides of the pathological spleen.
E. J. H. Birch

541. **Ion-exchange chromatography of inosine phosphates.** A. Deutsch and R. Nilsson (*Acta Chem. Scand.*, 1953, **7** [9], 1288–1292).—The method of Cohn and Carter for separating adenosine phosphates on a column of Dowex-1 resin (*J. Amer. Chem. Soc.*, 1950, **72**, 4273) is applied to the separation of mixtures of inosine and adenosine phosphates, orthophosphate and pyrophosphate, by means of Dowex-2 resin. Inosine monophosphate and adenosine diphosphate, and inosine diphosphate and adenosine triphosphate, are eluted by the same solvents; with the second pair of compounds the Cohn and Carter method needs modification to achieve separation. Recovery of individual compounds is at least 95 per cent.
C. E. Searle

542. **Analysis of adenosine triphosphate (ATP) and adenosine diphosphate (ADP) preparations by paper ionophoresis.** H. E. Wade and D. M. Morgan (*Biochem. J.*, 1954, **56** [1], 41–43).—A method is described for the separation of ATP, ADP, adenosine 5-phosphate, orthophosphate and pyrophosphate by paper ionophoresis. The ionophoresis technique differs from those normally used in requiring no reservoirs of buffer at the electrodes. The method has been used in the analysis of 7 samples of ATP and 1 of ADP.
B. Viney

543. **Studies on glycoproteins of the domestic fowl. I. Modification of the method of Elson and Morgan for the determination of hexosamine, and its applicability to tissue hydrolysates.** P. A. Anastassiadis and R. H. Common (*Canad. J. Chem.*, 1953, **31** [11], 1093–1107).—The method of Elson and Morgan (*Biochem. J.*, 1933, **27**, 1824) for the determination of hexosamine is studied spectrophotometrically for its application to the analysis of tissue hydrolysates. Interference from humin can be corrected for by appropriate blanks. It is necessary (*i*) to increase the strength of the buffer solution in order to stabilise the pH value at about 9·5, and, in extreme cases, first to neutralise the dried hydrolysate and (*ii*) to increase the concentration of acetylacetone reagent to 4 per cent. These modifications also reduce interference from hydroxyproline to a minimum. The use of an appropriate excess of acetylacetone depresses the formation of the secondary chromogen during acetylation. A procedure based on these findings is proposed for the direct determination of hexosamine in acid hydrolysates of tissues.
I. Jones

544. **The investigation of carbohydrates in proteins.** J.-M. Landucci, J. Pouradier and M. Pimont (*Bull. Soc. Chim. France*, 1953, **20** [11–12], 1072–1073).—The colour developed with thiobarbituric acid and hexoses in strong acids has an absorption max. at 446 mμ owing to formation of 5-hydroxymethylfurfural with the same max. Gelatin gives absorption max. at 455, 534 and sometimes at 430 mμ possibly due to a related substance. The natural occurrence of this related substance interferes with hexose determinations as 5-hydroxymethylfurfural. Absorption curves are given for gelatin, casein, ovalbumin, zein and haemoglobin.
E. J. H. Birch

545. **Microanalytical adaptation of Nessler's reaction. Application to biological media. II. Determination of 5 to 10 μg of nitrogen.** M. Herbain (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1233–1241).—A digestion apparatus, micro-heater, and micro-distillation apparatus are described in which the sample is evaporated, "mineralised," distilled into dil. acetic acid, the N being determined colorimetrically by Nessler's reagent. Necessary precautions are described. For 5 to 10 μg of N the error is ± 5 per cent.
E. J. H. Birch

546. **Reaction of naphthaquinone-4-sulphonate with amino-acids.** W. Troll (*J. Biol. Chem.*, 1953, **202** [1], 479–485).—Proline and hydroxyproline react quant. and stoicheiometrically with naphthaquinone-4-sulphonate (**I**) and a method is described for their determination in the presence of amino-acids. **I** also reacts with sarcosine, tryptophan, aniline and N-methylaniline. Protein hydrolysate (1 ml) containing hydrolysed protein (3–10 mg) and HCl (> 3 mol. equiv.) is set aside with 6 *M* $NaNO_2$ (1 ml) and glacial acetic acid (0·3 ml) for 1 hr., then

conc. HCl (10 ml) is added and the soln. is evaporated to 2 ml. The addition of HCl and the evaporation is repeated twice and the soln. is made alkaline to phenolphthalein with 2 *N* NaOH and then neutralised with 0·1 *N* HCl. Addition of *M* $NaHCO_3$ (1 ml), 0·02 *M* **I** (2 ml) and H_2O (to 10 ml) and, after 10 min., acetate buffer (pH 5) (1 ml) and 0·1 *M* ascorbic acid (1 ml) and water to 25 or 50 ml gives a soln. whose optical density at 480 mμ is determined; the optical density of a blank (omitting **I**) is deducted and the optical density is compared with that of a standard prepared with proline or hydroxyproline. The above procedure is applied to the analysis of lactoglobulin, bovine serum albumin and gelatin hydrolysates; the results are in good agreement with determinations made by other methods. Creatine forms an interfering colour with **I** and nitrosated creatine and sarcosine are inseparable by paper chromatography with benzyl alcohol - phenol (1 : 1) saturated with H_2O, the spots being detected with **I** in 0·1 *M* $NaHCO_3$.

R. J. BRIDGWATER

547. **Optical and enzymatic characterisation of amino acids.** J. P. Greenstein, S. M. Birnbaum and M. C. Otey (*J. Biol. Chem.*, 1953, **204** [1], 307–319).—The L- and D- isomers of 46 amino-acids of high optical purity were characterised by their mol. rotations in 5 *N* HCl and in glacial acetic acid solutions, and by their susceptibility to *Crotalus adamanteus* L-amino, acid oxidase and hog kidney D-amino-acid oxidase, respectively. Often the absolute magnitude of the optical rotations was higher in glacial acetic acid than in 5 *N* HCl. All the amino-acids were optically stable in glacial acetic acid except aminophenylacetic acid, which racemised under conditions in which its analogue amino*cyclo*hexylacetic acid retained its optical activity. With the exception of threonine, tryptophan, hydroxyproline and β-phenylserine all the L-amino-acids studied had more positive rotations in glacial acetic acid than in water. Determination of the mol. rotations of several glycyl-L-amino-acids showed that they all possessed more positive values in glacial acetic acid than in water. The effect on the mol. rotation of the L-amino acid residue by the introduction of the N-glycyl radicle was calculated for 3 solvents. Among the homologous series of straight chain L-amino-acids from C_3–C_{12} an optimal rate of oxidation by L-amino-acid oxidase occurred at about C_6. A comparison is drawn with a similar optimum that occurs in the relative rates of hydrolysis of the N-acetylated derivatives of these same L-amino-acids by hog kidney acylase-I.

P. N. CAMPBELL

548. **Quantitative determination of amino-acids in paper chromatograms. II.** E. Hiller and F. Zinnert (*Biochem. Z.*, 1953, **324** [2], 93–95).—A method previously described (*Brit. Abstr. C*, 1953, 266) is applied to the investigation of products of hydrolysis of albumins and to naturally occurring mixtures of amino-acids with satisfactory results. The method was used to determine the free amino-acids in human serum.

A. J. MEE

549. **Catalytic activity of cysteine and related compounds in the iodine - azide reaction.** D. W. Whitman and R. McL. Whitney (*Anal. Chem.*, 1953, **25** [10], 1523–1527).—The catalytic activity of cysteine and related compounds in the evolution of N by the iodine - azide reaction is described. Measurement of the N in Warburg respirometers at pH 4·63 showed that the evolution is quant. related to the mercaptans and disulphides present in the sample and is also dependent on the time of reaction, temp., reagent concn. and the group attached to the mercapto group. Evidence is also given as to the mechanism of the reaction. The reaction under the conditions investigated is not suitable for the measurement of mixtures of mercapto groups without further study, but is appropriate for the measurement of mercapto and disulphide groups attached to known radicles at the concn. encountered in biological fluids.

G. P. COOK

550. **Single dimension chromatographic separation of thyroxine and tri-iodothyronine.** E. C. Albright, F. C. Larson and W. P. Deiss (*Proc. Soc. Exp. Biol. Med.*, 1953, **84** [1], 240).—The two-dimensional chromatographic separation of thyroxine and tri-iodothyronine described by Gross and Pitt-Rivers (*Lancet*, 1952, *i*, 439) is time-consuming and the spots lack definition. The method described gives good separation of the two amino-acids, as well as of 3 : 5-di-iodothyronine and di-iodotyrosine. The separation was also followed by means of tri-iodothyronine and thyroxine labelled with ^{131}I.

Procedure—Cut Whatman No. 3 MM filter-paper into strips 29 cm by 7·2 cm, tapering to about 4·5 cm in 20 cm, leaving the remaining 7 cm or so as a narrow wick. Pipette about 50 μl of solution, containing about 1 mg of amino-acid per ml, along a horizontal line about 2 cm long, about 9 cm from the end of the wick. Suspend the strips so that the wick is submerged 1 to 2 mm in the developing solvent, which may be either a mixture of 44 ml of water dissolved in 125 ml of 2 : 4 : 6-collidine or a mixture of 80 ml butanol, 20 ml dioxan and 100 ml of 2 *N* aq. NH_3; this should be well shaken and the aq. layer discarded. Surround the system with a jar containing a small vessel of strong aq. NH_3 and develop the chromatograms at a const. temp. for 18 hr. Remove the strips, dry them at ≃ 80° C, spray them with 2·5 per cent. aq. Na_2CO_3 and dry them again. Finally spray with Koessler and Hankes diazo reagent prepared as follows. To a 50-ml flask immersed in an ice-bath add 1·5 ml of a soln. containing 0·9 g sulphanilic acid and 9 ml conc. HCl per 100 ml, and then 1·5 ml of a 5 per cent. $NaNO_2$ soln. Set aside for 5 min. and add an additional 6 ml of $NaNO_2$ soln.; after 5 min. dilute to volume. This soln. keeps several days in a refrigerator. Thyroxine, tri-iodothyronine and di-iodothyronine zones appear pink, while those of tyrosine and di-iodotyrosine are orange. The R_F values of the five compounds with the two reagents are given.

D. C. M. ADAMSON

551. **Calorimetric estimation of catalase activity.** H. D. Landahl (*Proc. Soc. Exp. Biol. Med.*, 1953, **84** [1], 75).—The amount of heat liberated by the catalytic decomposition of H_2O_2 can be used as a measure of catalase activity; the method is rapid and simple and particularly convenient for the examination of blood.

Procedure—To a plastic 15-mm × 9-cm test tube about 2 g in weight add 1 drop of capryl alcohol and 5 ml of an aq. soln. containing 0·5 ml of 30 per cent. H_2O_2 and 1 ml of neutral phosphate buffer (equal parts of 0·1 *M* Na_2HPO_4 and 0·04 *M* NaH_2PO_4). Insert a thermometer in the tube and place the tube in a test-tube holder made of a block of wood with holes 8 cm deep by 1·8 cm diameter, in such a manner as to ensure minimum contact with the wood. Set aside at a const. temp. for some time so that the temp. of the solution is 24·5° to 25·5° C. Pipette 0·01 ml of blood on to

the bulb of a clinical thermometer (scale range 33° to 43° C), note the temp. on the original thermometer, and stir the contents of the tube with the clinical thermometer for 15 sec. Read the thermometer after 8 min. The average temp. rise for human blood was found to be 12° C corresponding to a decomposition of 14 g of H_2O_2 for each ml of blood. Comparisons of the time course of the liberation of heat with that of the evolution of O, and the disappearance H_2O_2 show good agreement.
D. C. M. ADAMON

552. **Colorimetric method for the estimation of catalase.** F. Patti and P. Bonet-Maury (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1177–1180).—To 3 ml of standardised H_2O_2 (10 vol.) are added 3 ml of a soln. of the catalase diluted 10^{-4} in a phosphate buffer at pH 7, and the whole is maintained at 19° C. One ml is removed by a pipette after predetermined time intervals and put into a photometer cell containing 4 drops of conc. H_2SO_4 and 2 drops of Ti reagent, and the optical density is measured at 450 mμ. The reaction is nearly complete after 90 min., but the H_2O_2 decomposition after a given time between 20 and 90 min. varies linearly with concn. of catalase. The change of catalytic activity with time confirms other observations that there are two components in catalase, one labile leading to a rapid reaction and the other stable leading to a slow reaction. E. J. H. BIRCH

553. **Ultramicrospectrophotometric determination of cytochrome oxidase for quantitative histochemistry.** H. H. Hess and A. Pope (*J. Biol. Chem.*, 1953, **204** [1], 295–306).—A rapid, sensitive, ultramicroassay method for cytochrome-oxidase activity, based upon direct spectrophotometric observation of the rate of oxidation of reduced cytochrome-*c*, is described. The high sensitivity is due to the use of an enzyme prep. clarified by the action of Na deoxycholate and to a relatively high concn. of cytochrome-*c* soln. providing an excess of cytochrome during the first 2 to 3 min. of the reaction. The method measures the oxidase activity in less than 0·2 μg dry wt. of rat heart in 70 μl of test soln. The method has been used to measure the cytochrome-oxidase activity in rat heart, kidney cortex, liver, spleen, skeletal muscle, cerebral cortex and cerebellum. The relative activities of these tissues found by the proposed method are comparable with those reported by other investigators.
P. N. CAMPBELL

Drugs

554. **Separation of alkaloids by paper electrophoresis.** W. Deckers and J. Schreiber (*Naturwissenschaften*, 1953, **40** [21], 553–554).—Separation of alkaloids by electrophoresis by means of Grassmann apparatus is described. The soln. containing alkaloids is placed on filter-paper, the paper is sprinkled with a suitable buffer solution and suspended in the apparatus for 2 to 5 hr. during which time the distance between the substances reaches 3 to 10 cm. The separation can be performed continuously, $\simeq$ 100 mg of an alkaloid mixture being quant. separated in 40 hr.
S. K. LACHOWICZ

555. **Separation of alkaloids from their N-oxides by microchromatography on paper.** R. Munier (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1225–1231).—Separation between alkaloids and their N-oxides by means of *n*-butanol - acid mixtures is poor, but separation can be effected by means of mixtures of acetone (75 ml) - dil. aq. NH_3 (25 ml) for strychnine, morphine, atropine and scopolamine and their N-oxides; *n*-propanol (75 ml) - 5 per cent. aq. acetic acid (25 ml) is suitable for separating eserine and geneserine. A more satisfactory separation is made by using as solvents *n*-, *iso*- and *sec*.-butanol, saturated with water and *tert*.-butanol, *n*- and *iso*-propanol with water (3:1 vol. ratio), and with paper "salted" by treatment with 0·067 *M* to *M* soln. of KH_2PO_4, wiping, and drying for 48 hr. R_F are quoted and the following conditions are recommended: for strychnine - genostrychnine, 0·5 *M* phosphate paper, *n*-propanol solvent; for morphine - genomorphine, 0·1 *M* paper, *sec*.-butanol; for atropine - genatropine and for scopolamine - genoscopolamine, 0·2 *M* paper, *iso*-butanol; and for eserine - geneserine, 0·2 *M* paper, *n*-butanol. The chromatography is effected by the ascending technique with a solvent run of 35–40 cm, and the spots are developed with iodide - bismuth reagent with acetic or tartaric acid. E. J. H. BIRCH

556. **Photometric method of determination of small quantities of yohimbine.** J. Kolšek (*Z. anal. Chem.*, 1953, **140** [3], 186–188).—Photometric determination of yohimbine hydrochloride, after oxidation with H_2O_2 or $NaNO_2$, is described. The use of H_2O_2 is preferred as NO_2' in solution interferes with the measurements. With either, the use of very dilute soln. of yohimbine is recommended as only then is Beer's law valid. S. K. LACHOWICZ

557. **Determination of caffeine in cola seeds.** J. A. C. Van Pinxteren and G. Schallenberg-Heertjes (*Pharm. Weekbl.*, 1953, **88** [47–48], 805–808).—The powdered seeds (6 g) are shaken for 1 hr. with 60 g of $CHCl_3$ and 6 ml of aq. NH_3. The residue obtained by evaporation of 10 g of the filtered $CHCl_3$ extract (≡ 1 g of seed) is dissolved in warm water, and the solution is mixed with 25 ml of 0·1 *N* I soln., 5 ml of 4 *N* H_2SO_4 and 20 ml of saturated NaCl, made up with water to 100 ml, and set aside for 5 min. After filtration through asbestos the first 20 ml is rejected, and 50 ml of the filtrate are back-titrated with 0·1 *N* $Na_2S_2O_3$. The no. of ml of 0·1 *N* I soln. consumed multiplied by 0·00485 gives the wt. of anhyd. caffeine.
P. S. ARUP

558. **Infra-red analysis of active ingredients in ointments. II. Pilocarpine hydrochloride and phenacaine hydrochloride.** W. H. Washburn (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [11], 698–699). A quant. method for determining pilocarpine and phenacaine HCl by i.r. analysis is described. Phenacaine HCl (1 per cent. in a 1-g sample) and pilocarpine HCl (2 per cent. in a 1-g sample) can be determined with an accuracy of ± 4 per cent. The method is specific. N. M. WALLER

559. **The analysis of digitoxin tablets.** D. Banes and J. Carol (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [11], 674–678).—Liquid - liquid partition chromatography is used for the quant. separation of digitoxin, the recovery of gitoxin and the detection of digitoxigenin in digitoxin tablets. Formamide - water adsorbed on Celite is used as the immobile phase and C_6H_6 - $CHCl_3$ is the mobile solvent. The substances are determined colorimetrically by the Keller - Kiliani and Raymond reactions. The method has been applied successfully to the assay of commercial digitoxin tablets.
N. M. WALLER

560. **Non-aqueous titration of antibiotic substances.** C. N. Sideri and A. Osol (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [11], 688–689).—Aureomycin, Ilotycin, Magnamycin and Terramycin as the base or HCl salt can be quant. titrated in soln. in glacial acetic acid with a soln. of $HClO_4$ in dioxan. The same method can be applied to ointments and suppositories containing these antibiotics, after digestion with $CHCl_3$ at room temp. for 30 min.
N. M. Waller

561. **Paper electrophoresis of streptomycins.** M. C. Foster and G. C. Ashton (*Nature*, 1953, **172**, 958–959).—A method is described for the separation of streptomycin, mannosidostreptomycin and allied substances by paper electrophoresis. Spray reagents diacetyl, naphtharesorcinol and alcoholic KOH mixed with 1 per cent. acetylacetone in redistilled *n*-butanol are all suitable for detecting small quantities of streptomycin on paper. The test solutions are applied to the paper, which is then wetted evenly with a buffer solution of pH 5·0. After passage of the current for 16 hr. the paper is removed and dried, and either sprayed with one of the above reagents to develop coloured spots or laid on an agar plate seeded with a suitable organism.
N. M. Waller

562. **Studies on penicillinase. Spectrophotometric determination of penicillinase.** P. Baudet and E. Cherbuliez (*Helv. Chim. Acta*, 1953, **36** [7], 2055–2064).—A method for determining penicillinase is described that is based on the fact that, compared with its degradation product benzylpenicilloic acid, benzylpenicillin has a much greater absorption at 240 mμ; this decreases progressively in presence of penicillinase to 262·5 mμ, but that of benzylpenicilloic acid is approx. the same as that at 240 mμ. The extinction coefficient at 262·5 mμ subtracted from the extinction coefficient at 240 mμ is thus a direct measure of the quantity of benzylpenicillin remaining in the solution.
A. Storfer

563. **Determination of the lipase activity of pancreatin.** M. Pesez and R. Willemart (*Ann. Pharm. Franç.*, 1953, **11** [9–10], 608–614).—A 60-mg sample of pancreatin is finely ground with 5 g NaCl and a paste is formed and made up to 200 ml with 7·5 per cent. NaCl soln. To a 1-ml aliquot are added 10 ml of glycine - NaOH buffer (pH 9·4), 2 ml of Na cholate solution (as emulsifier), 7 ml of water and 1·5 ml of purified tributyrin. The mixture is shaken and maintained for 2 hr. at 40° C and, after addition of neutral formaldehyde and ethanol, is titrated with alcoholic 0·2 *N* KOH. The indicator used is phenolphthalein (2 g) with thymol blue (0·1 g) in 100 ml of ethanol. A blank determination is carried out with pancreatin inactivated on a bath of boiling water. The relationship between weights of a sample of pancreatin and vol. of 0·2 *N* KOH used, and the effect of time of digestion are investigated.
E. J. H. Birch

564. **The chemical assay of adrenal cortex extracts.** D. Banes (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [11], 669–673).—The separation and quant. recovery of mg quantities of the active 11-oxygenated corticosteroids is accomplished by adapting the systems of Burton, Zaffaroni, and Keutmann (*J. Biol. Chem.*, 1951, **188**, 763) to liquid - liquid partition chromatography on Celite. The steroids are determined colorimetrically and identified by i.r. spectrophotometry. Calculated glycogenic potencies are in good agreement with bio-assay results.
N. M. Waller

565. **The liquid - liquid counter-current separation of menthol - menthyl acetate mixture.** J. V. Swintosky, R. Kuramoto, and T. Higuchi (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [11], 666–668).—The results are presented of the application of a simple funnel assembly in the investigation of the use of the general counter-current distribution method in volatile oil systems (menthol - menthyl acetate mixture). The method is preferable to fractional crystallisation or distillation since the choice of immiscible solvents ensures a reasonable difference of partition coefficients; similarity of b.p. or of m.p. is no disadvantage. Results are within experimental error compared with theory and it is calculated that with complete recovery of the solute pair 99 per cent. separation can be effected.
N. M. Waller

566. **Identification of derivatives of barbituric acid by X-ray analysis. VII. Enphenemal and binary mixtures of Enphenemal with other barbiturates.** E. J. Hansen and B. Jerslev (*Dansk. Tidsskr. Farm.*, 1953, **27** [12], 261–265).—By means of the technique of Huang (*Acta Pharm. Intern.*, 1951, **2**, 43, 95, 173, 289, 317, and 361) the monomorphous character of Enphenemal (methylphenobarbitone) is confirmed (*cf.* Kofler *et al.*, "*Mikro-Methoden zur Kennzeichnung organischer Stoffe und Stoffgemische*," Innsbruck, 1948). The m.p. curve for mixtures of Enphenemal and Enhexymal (a similar 1:5:5-trisubstituted derivative of barbituric acid) shows that the two substances form mechanical mixtures only. In finely powdered mixtures of the two drugs, the min. amount of either detectable by X-ray analysis is $\simeq$ 10 per cent.
P. S. Arup

567. **Titration of mixtures of trimethadione and phenobarbitone or phenylmethylbarbitone.** H. Vasbinder and H. R. Van Der Sijde (*Pharm. Weekbl.*, 1953, **88** [47–48], 801–804).—Budde's titration of the barbituric drugs with 0·1 *N* $AgNO_3$ in presence of Na_2CO_3 (*Brit. Abstr. B*, 1935, 45) is unaffected by the presence of trimethadione. Mixtures may be analysed by applying the above method, in addition to the U.S.P. XIV method (alkalimetric hydrolysis) with thymolphthalein as indicator. This latter method gives a measure of the total trimethadione and barbituric drugs.
P. S. Arup

568. **Detection of some *iso*nicotinic acid and nicotinic acid derivatives on filter-paper.** F. Leuschner (*Naturwissenschaften*, 1953, **40** [21], 554).—Several methods of detecting *iso*nicotinic acid and nicotinic acid deriv. by the spot analysis of < 6-μg amounts are outlined. A concn. of *iso*nicotinic acid hydrazide as small as 0·5 μg per ml can be detected by the following procedure: 1 ml of neutral eluate from paper chromatography is mixed with 1 ml of 4 per cent. aq. CNBr soln. and heated for 5 min. on a bath of boiling water. After addition of 0·5 ml of 32 per cent. NaOH soln. the fluorescence produced is examined in a fluorimeter embodying a Pulfrich photometer. The Lambert - Beer law is obeyed only for concn. up to 25 μg per ml.
S. K. Lachowicz

569. **Biochemical and micro-analytical study of some tuberculostatic hydrazides.** C. Duval, N. P. Buu-Hoï, N. D. Xuong and M. Jacquinot (*Mikrochim. Acta*, 1953, [3], 212–219).—As the antitubercular property of certain hydrazides appears to be due to their ability to form complexes with metals the use of these hydrazides as micro-analytical reagents is suggested. Accordingly the effect of 8 active reagents—salicylic acid hydrazide, 5-chloro-, 3:5-dichloro-, 5-bromo-, 3:5-dibromo- and

3:5-di-iodo-salicylic acid hydrazides, nicotinic acid hydrazide and *iso*nicotinic acid hydrazide—on solutions of certain ions is noted, particularly for $Cu^{\cdot\cdot}$, $V^{\cdot\cdot\cdot}$, and $Ce^{\cdot\cdot\cdot\cdot}$. For $Cu^{\cdot\cdot}$, only 5-chlorosalicylic acid hydrazide gives a ppt. in the cold; then this is only for $CuSO_4$. Thermolysis curves are given for the Cu complexes of salicylic acid hydrazide and its 5-chloro and 3:5-dichloro deriv. and for those deriv. of nicotinic acid hydrazide, from which the formula of the complexes is deduced. The estimation of Cu with 5-chlorosalicylic acid hydrazide is then described, and found capable of determining quantities of $\simeq$ 1 mg with an error of 1 to 1·2 per cent.

J. H. WATON

570. **Qualitative and quantitative analysis of mixtures of sulphonamides. VII.** P. L. de Reeder (*Anal. Chim. Acta*, 1953, **9** [4], 314–323).—Estimations of therapeutically useful sulphonamides alone and in mixtures by the bromometric methods of Wojahn and Conway and by the nitrite titration of the free aromatic-NH_2 group are compared. Conditions and percentage recoveries are listed. E. J. H. BIRCH

571. **Determination of Largactil in biological fluids. A study of its passage through the animal organism.** P. Dubost and S. Pascal (*Ann. Pharm. Franç.*, 1953, **11** [9–10], 615–619).—Largactil [chlorpromazine; 3-chloro-10-(3-dimethylaminopropyl) phenothiazine] is distinguished from similar compounds by a carmine red colour produced with conc. H_2SO_4, orange-red with H_2SO_4 and CrO_3, winelees red with $PdCl_2$, and a pale yellow ppt. with NaBrO. The solution in ethanol gives a pomegranate red colour with Br water, and on making the solution alkaline a white ppt. sol. in methanol is obtained. Free chlorpromazine in blood (5 ml) is determined by making the blood alkaline with 60 per cent. KOH, diluting, and extracting 3 times with ether. The ether extracts are extracted with 0·1 *N* H_2SO_4 and after removal of ether an equal vol. of conc. H_2SO_4 is added to an aliquot and the colour is measured in a photometer (Meunier; screen 55). The combined chlorpromazine is determined similarly after hydrolysis with HCl before ether extraction. Free chlorpromazine in urine is determined as for blood with larger quantities. Combined chlorpromazine in urine is determined after previous hydrolysis (alkaline hydrolysis—refluxing with 20 per cent. NaOH for 2 hr.—is preferable for rabbit urine and acid hydrolysis with HCl for dog urine). Oxidation products in rabbit urine give a violet tint to the final colour, but this colour can be avoided by diluting the acid extraction liquor before adding the conc. H_2SO_4. Determination of chlorpromazine in blood and urine after administration to dogs and rabbits gives a low recovery indicating considerable degradation in the organism.

E. J. H. BIRCH

572. **Estimation of cholesterol and triterpenols in unsaponifiable fraction of wool wax.** H. Duewell (*Anal. Chem.*, 1953, **25** [10], 1548–1550).—Solutions are prepared in alcohol-free chloroform. Three 0·3-ml aliquots are taken from each sample and placed in 100-ml flasks, which are stoppered with cellophane covered corks. Ten ml of alcohol-free $CHCl_3$ is added followed by 5 ml of Liebermann-Burchard reagent (1 vol. conc. H_2SO_4 + 9 vol. acetic anhydride). The soln. are stored in the dark and their absorbances are measured at 460 mμ and 620 mμ after 30 min. The percentages of cholesterol and triterpenol are calculated from these measurements, the absorbances of cholesterol and triterpenol being previously determined at the two λ used. Results are within $\pm$ 3 per cent.; wool-wax alcohols do not interfere. The smallest amounts that can be determined to the reported accuracy are 60 μg of cholesterol and 10 μg of triterpenol.

G. P. COOK

Food

573. **Ultra-violet spectrophotometric estimation of the quality of mineral oils extracted from bread.** M. A. Cookson, J. B. M. Coppock and R. Schnurmann (*Analyst*, 1953, **78**, 695–701).—In the method suggested the unsaponifiable fraction of the total oils extracted from the bread is treated with H_2SO_4 (85 per cent. w/w) at 50° C, the natural saponifiable oil being thus destroyed without completely removing the unsaturated hydrocarbon constituents of mineral oil refined to different degrees. The recovered mineral oil is then examined spectrophotometrically to determine its quality, and a criterion based on the absorption at 260 mμ is suggested for the quality of mineral oil suitable for use as lubricant in bread-making plant. Some properties of the natural unsaponifiable oils of bread are described. A. O. JONES

574. **The chromatographic determination of glutamic acid in wheat gluten and gluten hydrolysates.** P. Morries and R. E. Stuckey (*Analyst*, 1953, **78**, 636–640).—In the method described the total dicarboxylic acids (glutamic and aspartic) are adsorbed on Amberlite IR-4B resin (or Zeocarb De-acidite E) at pH 3 to 4 and eluted with *N* HCl. After evaporation of the eluate to low bulk *in vacuo*, the amino nitrogen is determined by the copper method or the total N by the Kjeldahl method. The aspartic acid present in the original sample is then determined by means of descending paper chromatography with a phenol-water system and a ninhydrin spray. The colour from the spots is extracted with aq. pyridine and measured spectrophotometrically at 570 mμ and the N equiv. to the aspartic acid is deducted from the total N found, the glutamic acid being found by difference.

A. O. JONES

575. **The "albuminoid ammonia value" in the analysis of fruit juices, squashes and cordials.** S. N. Mitra and S. C. Roy (*Analyst*, 1953, **78**, 681).—The procedure for determination of the albuminoid ammonia value of vinegar (Mitra, *Brit. Abstr. C*, 1953, 481) has been applied with a slight modification to soft drinks and serves as a convenient sorting test for detection of deficiency in fruit-juice content. To counteract the reducing action of sugar in the sample, solid $KMnO_4$ as well as the usual alkaline $KMnO_4$ solution must be added before the second stage of the distillation. A. O. JONES

576. **Determination of potassium in wine. Contribution to the new edition of official rules.** O. Reichard (*Z. anal. Chem.*, 1953, **140** [3], 188–197).—Three methods of determination of K in wine by means of wine-ash analysis are described. In these methods K is pptd. and determined gravimetrically as $KClO_4$, the 5-nitrobarbituric acid salt or as $[(C_6H_5)_4B]K$. The last method can be also used for direct determination of K + NH_4 + org. bases content or K + org. bases content in wine with great accuracy. S. K. LACHOWICZ

577. **An adulterant of dried sage.** D. F. H. Button (*Analyst*, 1953, **78**, 679).—A foreign leaf found in samples of dried sage is believed to be that of a species of *Cistus*, a flowering shrub common

in the Mediterranean region. It is distinguished microscopically from sage by its stellate hairs (illustrated) and presence of Ca oxalate crystals.
A. O. JONES

578. **A colorimetric method for the determination of vitamin A and carotene by perchloric acid.** P. Flesch (*Proc. Soc. Exp. Biol. Med.*, 1953, **84** [1], 148).—The method is based on the measurement of a purple-red colour (max. 525 to 530 mμ) produced from vitamin A by reaction with a mixture of hydrochloric and perchloric acids in pentyl acetate. Under the same conditions carotene gives a bluish-green colour with max. absorption at 725 to 760 mμ. In a mixture of vitamin A and carotene the absorption at 725 to 760 mμ gives the carotene content; the absorption at 525 mμ is corrected for the absorption due to carotene before converting to vitamin A. Beer's law is obeyed for up to 30 μg of both vitamin A and carotene under these conditions. Vitamin D gives no colour. The perchloric acid reagent is prepared by diluting 3 vol. perchloric acid (70 to 72 per cent.) with 1 vol. distilled water. *Preparation of standard curves.*—Measure 0·1 ml of a chloroform solution of vitamin A (300 μg per ml) or carotene (300 μg per ml) into a test tube, add 1·6 ml pentyl acetate and 0·4 ml conc. HCl. Shake the tube and set it aside at room temp. for 4 to 5 min., and then add 2 ml of perchloric acid reagent. Add 0·3 to 0·4 ml absolute alcohol with vigorous shaking until the layers become miscible, and 5 to 15 min. later measure the extinction of each soln. at 525 to 530 mμ (vitamin A and carotene) or 725 to 760 mμ (carotene), using a similar mixture containing 0·1 ml chloroform in place of the standard solution as a blank. Draw three standard curves relating (*i*) extinction of soln. at 525 to 530 mμ and amount of vitamin A, (*ii*) extinction of soln. at 525 to 530 mμ and amount of carotene, (*iii*) extinction of soln. at 725 to 760 mμ and amount of carotene. *Unknown samples*—Follow the same procedure as for preparing the standard curves; when carotene alone is present take readings at 725 to 760 mμ only; for vitamin A alone take readings at 525 to 530 mμ and read the amount of carotene or vitamin A from the appropriate standard curve. When both vitamin A and carotene are present determine the amount of carotene from the extinction at 725 to 760 mμ and calculate the extinction at 525 to 530 mμ which is due to carotene. Subtract this from the observed extinction at 525 to 530 mμ and read off from the standard vitamin-A curve the vitamin-A content.
D. C. M. ADAMSON

579. **Adsorption of thiamine on glassware.** K. T. H. Farrer and W. C. J. Hollenberg (*Analyst*, 1953, **78**, 730–731).—In some circumstances quinine sulphate (used as reference standard in thiamine determinations) can be adsorbed on the walls of glassware, but this can be prevented by first treating the glassware with boiling 30 per cent. NaOH soln. (Bird, *N.Z. J. Sci. Tech.*, 1949, **30**, 344). Although Bird claims that thiochrome is not adsorbed, it is now found that thiamine is, and that the loss may be 21 to 44 per cent. according to the number and type of vessels used. Bird's method of preventing adsorption of quinine sulphate also prevents adsorption of thiamine, but the preventive action of alkali treatment is not permanent. With strongly acid solutions there is no adsorption of thiamine, but, unless the alkali treatment is applied, fluorescent substances, shown not to be thiochrome, are removed from the glass by the mixture of ethanol and butanol. The investigation has been restricted mainly to glassware from one maker. A. O. JONES

580. **Paper chromatography of choline and the vitamins B_1, B_2, niacin and niacinamide. Preparation of radioactive choline acetate and study of its hydrolysis.** A. Heyndrickx (*J. Amer. Pharm. Ass., Sci. Ed.*, 1953, **42** [11], 680–681).—A qual. determination of these substances by paper chromatography with *n*-propanol - hydrochloric acid as solvent is described. Spot tests for each are given sensitive to 0·5 μg of vitamin B_1, 0·1 μg of vitamin B_2, 10 μg of niacin, 5 μg of niacinamide and 10 μg of choline. The prep. of radioactive choline acetate is included. After 24 hr. under atmospheric conditions choline acetate hydrolyses to the extent of 54 per cent. and after 3 days to 61 per cent. In an aq. medium hydrolysis is complete in a few sec.
N. M. WALLER

581. **Microbiological micro-estimation of the citrovorum factor.** M. Polonovski and G. Lévy (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1167–1169).—The method is standardised by adding definite quantities (0·05 to 1·0 μg) of pure leucovorine to 1 ml of a culture medium (detailed), treating, after rapid sterilisation, with *Leuconostoc citrovorum* and estimating nephelometrically after 20 hr. at 37° C. The estimation is carried out similarly. This method was applied to normal and pathological urine, and to investigate the activity (found to be nil) of cortisone in replacing the factor.
E. J. H. BIRCH

582. **Determination of total phosphatide in commercial lecithin.** H. H. Hutt, H. Weatherall and T. Culshaw (*Analyst*, 1953, **78**, 712–716).—In a method previously reported (Hutt *et al.*, *Analyst*, 1944, **69**, 39) the phosphatide content of commercial lecithin is assessed by means of the difference between the petrol-insol. and the acetone-insol. matter. A modified method with improved precision for determination of the acetone-insol. matter is now presented. The difference between the acetone-insol. matter so determined and the petrol-insol. matter is suggested as giving a suitable measure of the phosphatide content. A. O. JONES

Agriculture and Plant Biochemistry

583. **A new method for the quantitative determination of chloride in plant material.** S. Samson (*Nature*, 1953, **172**, 1042).—An electrometric method based on the Ag - AgCl electrode is briefly described.
E. G. BRICKELL

584. **Determination of ammonium, amide, nitrite and nitrate nitrogen in plant extracts.** J. E. Varner, W. A. Bulen, S. Vanecko and R. C. Burrell (*Anal. Chem.*, 1953, **25** [10], 1528–1529).—Ammonium, amide, nitrite and nitrate N are determined in plant extracts from a single portion of the sample. The extract, freed from proteins, is buffered at pH 10 with borate and is placed in a semi-micro Kjeldahl distillation unit; ammonium N is removed by vacuum distillation at 40° C. Conc. NaOH soln. (40 per cent.) is added and amide N is removed by steam distillation at 100° C. Addition of $FeSO_4$ (20 per cent. soln.) follows and the distillation is continued in order to remove nitrite N. The nitrate N is removed after addition of saturated Ag_2SO_4 soln. to catalyse reduction by $FeSO_4$. The amount of NH_3 in the receiver flask is determined each time by titration with 0·01 *N* H_2SO_4. Glucose and acetone interfere and a separate procedure involving the use of ion-exchange resins is given for extracts containing interfering substances. G. P. COOK

585. **The lipase activity of certain cereal products.** W. H. Templeton and B. R. Carpenter (*Analyst*, 1953, **78**, 726–727).—The method described for determination of lipase activity is a modification of that of Hutchinson *et al.* (*Brit. Abstr. C*, 1953, 219). The fat-freed sample (*e.g.*, malt flour) is incubated for 6 hr. with 7 drops of olive oil and 0·2 ml of water, after which the mixture of oil and fatty acid is extracted with light petroleum and weighed. The oil is then dissolved in a neutral alcohol - benzene mixture and the fatty acid content is determined by titration with 0·02 *N* NaOH. By assuming that the free fatty acid is oleic acid, the hydrolysed oil can be expressed as a percentage of the total oil, thereby affording an indication of the lipase activity of the flour and the consequent possibility of its conferring a soapy off-flavour on baked products. A. O. JONES

586. **Chromatographic determination of amino-acids of a cereal seed (barley), a hay, and a linseed cake.** J. P. Dustin, E. Schram, S. Moore and E. J. Bigwood (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1137–1147).—A barley reaped in July, a hay reaped in June containing 65 per cent. *Holeus lanatus* and 5 per cent. each of seven other species, and a linseed cake from linseed reaped in June (all grown in Belgium) were finely milled and sieved. A total analysis was made by standard (A.O.A.C.) methods. Samples are hydrolysed and chromatographed on Dowex-50 by the method described by Schram *et al.* (*Anal. Abstr.*, 1954, **1**, 148) and Dustin *et al.* (*Anal. Abstr.*, 1954, **1**, 343), and the results for each amino-acid from each foodstuff are summarised in graphs. Cystine is completely destroyed during the hydrolysis in presence of carbohydrates and must be estimated by previous oxidation to cysteic acid in another sample. The recoveries of N as protein by this chromatographic method is 85·5 per cent. for the barley, 78·5 per cent. for the hay and 93·9 per cent. for the linseed (without correction for humic N present). E. J. H. BIRCH

587. **Paper chromatography of some vegetable glycosides.** M.-M. Janot, E. Saïas and M. Foucher (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1101–1110).—Paper chromatographic separations of a number of glycosides and their aglycones are carried out by using as solvents: the two layers from the mixture of *n*-butanol (4 vol.) - acetic acid (1 vol.) - water (5 vol.) (org. phase, **I**, aq. phase, **II**); the org. phase from the mixture of ethyl acetate (2 vol.) - pyridine (1 vol.) - water (2 vol.), **III**; phenol saturated with water in an NH_3 atmosphere, **IV**; 15 per cent. v/v aq. acetic acid, **V**; 10 per cent. aq. NaCl, **VI**; *n*-butanol saturated with water, **VII**; and *n*-butanol (4 vol.) - ethanol (1 vol.) - water (5 vol.), **VIII**. R_F are quoted for calycanthoside, fraxoside, aesculoside, methylaesculoside and their aglycones with solvents **I** to **V**, for salicoside and populoside with **I** to **VI**, for coniferoside, syringoside, vanilloside and piceoside with **I**, **II**, **V**, **VI**, **VII**, for colchicoside with **I**, **III**, **IV**, **VI**, for gentiopicroside with **I**, **III**, **IV**, **V**, **VI**, for monotropitoside with **I**, **III**, **IV**, **V**, **VIII**, for aucuboside and asperuloside with **I**, **III**, **IV**, **V**, and for loroglossoside with **I**, **III**, **V**, **VI**. Aesculoside, methylaesculoside and their aglycones can be detected by their u.v. fluorescence and the former by Barton's reagent [$K_3Fe(CN)_6$ - $FeCl_3$ - HCl] for phenols. Salicoside and populoside, which can be hydrolysed by $Ba(OH)_2$ or emulsin, are detected by hydrolysis on the paper followed by treatment with Barton's reagent. Coniferoside, syringoside, vanilloside and piceoside are detected by their fluorescence under u.v. light; the aglycones of the first two react with Barton's reagent and of the last two with 2:4-dinitrophenylhydrazine. Colchicoside is detected by its violet-blue fluorescence under u.v. and gives a yellow colour with benzidine, and its aglycone (demethylcolchicine) gives a yellow fluorescence and a colour with Barton's reagent, as does demethylcolchicine which is found free in the plant. Gentiopicroside shows a brown spot with u.v. light at 2537 A but no colour with Wood's light, but the aglycone gives a bluish colour with both types of illumination; the glycoside gives a brown and the aglycone an orange-pink colour with benzidine, naphtharesorcinol gives a yellow colour, and the aglycone reacts with Barton's reagent. Monotropitoside yields various products with various methods of hydrolysis; an enzyme from *Monotropa* affords methyl salicylate and primaverose; emulsin has no effect, but 2 per cent. H_2SO_4 yields methanol, salicylic acid, glucose and xylose; alkaline hydrolysis yields methanol and salts of salicylic acid primaveroside. Naphtharesorcinol gives blue spots with these glycosides. Both aucoboside and asperuloside have aglycones that tend to polymerise unless hydrolysis is effected carefully with emulsin. The spots are detected with NH_3 - $AgNO_3$, with benzidine (grey-brown) or with aniline phthalate (brown). Asperuloside and its aglycone fluoresce under u.v. light at 2537 A and give a green colour with acids (*e.g.*, trichloroacetic acid which does not attack the paper is preferred). Loroglossoside is hydrolysed with trichloroacetic acid on the paper to its aglycone which can be detected by Barton's reagent. E. J. H. BIRCH

588. **Distinction between flavone-glycosides and flavones.** R. Leu and P. Hagedorn (*Z. anal. Chem.*, 1953, **139** [2], 96–98).—Colour tests are given for rutin, quercetin, morin, quercitrin and hyperoxide. The reagents used are: 0·01 *M* cupric acetate; (1 + 1) mixture of 0·01 *M* cupric acetate and 1 per cent. w/v SeO_2; a (1 + 1) mixture of 0·01 *M* cupric acetate and 10 per cent. w/v acetic acid; 0·01 *M* Cd acetate; a (1 + 1) mixture of 0·01 *M* Cd acetate with 1 per cent. w/v acetic acid. These are applied to a 1 per cent. w/v soln. in CH_3OH. A list of colours obtained with each reagent is given. G. P. COOK

See also Abstracts 489, 501, 502, 503, 504, 505, 596, 599, 612, 630.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

589. **The Garner heavy duty quartz fibre microbalance.** J. A. Kuck, P. L. Altieri and A. K. Towne (*Mikrochim. Acta*, 1953, [3], 254–265).—A description is given of the Garner quartz-fibre microbalance, which is suitable for Pregl ultramicro-analysis. A mounting to eliminate vibration is also described. The balance is designed to operate within a capacity range of 0·6 to 5·0 g, a weighing range of only 1·5 mg being covered by the fibre itself. Under a 5-g load, there is a strict proportionality between torsion and weight to within ± 0·001 mg. With a 0·4-g platinum combustion boat, the reproducibility of weighing with the torsion fibre under continuous strain is ± 0·26 μg. J. H. WATON

590. **Design and operating technique of a vacuum drying oven. I. Design of the oven.** S. D. Gardiner (*Analyst*, 1953, **78**, 709–711).—A symmetrical vacuum drying oven of large heat capacity and uniform temp. distribution has been designed. It has a shallow, hollow but thick metal base holding six sample dishes with the minimum of free space and is designed to operate efficiently from 50° to 90° C. For drying at atm. pressure the temp. may be raised to 105° C. A. O. JONES

591. **A vacuum valve to provide small controlled leak-rates.** R. Forman (*Rev. Sci. Instrum.*, 1953, **24** [4], 326–327).—The optically polished ends of two glass tubes are held in contact by means of a metallic outer jacket. Controlled heating of the outer jacket causes variation of the degree of contact of the glass surfaces. G. SKIRROW

592. **Oil manometer for ultra-high vacuum systems.** M. A. Biondi (*Rev. Sci. Instrum.*, 1953, **24** [10], 989–990).—A direct reading oil manometer for the pressure range 0 to 30 mm is described. The manometer, which contains a heating element for degassing purposes, is equipped with a vernier and has an accuracy of approx. ± 0·01 mm. G. SKIRROW

593. **A conduction-cooled trap for demountable vacuum systems.** J. Pollard (*Rev. Sci. Instrum.*, 1953, **24** [10], 996–997).—The trap consists of a baffle assembly of 32 semicircular copper sheets equally spaced inside a copper cylinder in such a way that there is no straight path through the cylinder. The baffles are cooled by a copper rod which dips into a flask of coolant. G. SKIRROW

594. **A high-pressure gas valve for use [at pressures] up to 3000 atmospheres.** D. W. Robinson (*J. Sci. Instrum.*, 1953, **30** [12], 483–484).—The valve spindle is in two parts separated by a flexible circular disc of tempered pen steel. Screwing in the spindle distorts the disc which, in turn, transmits motion to the second part of the spindle, thus causing the valve to close by forcing a spring-loaded steel ball into its seating. G. SKIRROW

595. **An automatic micro-manometer for the measurement of low air speeds.** R. A. K. Long (*J. Sci. Instrum.*, 1953, **30** [12], 481–482).—A two-liq., null-reading micro-manometer is described. The system is electrically driven to the balance point under the control of an electronic proximity switch. The apparatus has been used for reading the pressure at a pitot seven times per min., with an accuracy of ± 0·0005 in. of water. G. SKIRROW

596. **Separation of biological mixtures with a new counter-current partition apparatus.** A. Kepes (*Bull. Soc. Chim. Biol., Paris*, 1953, **35** [10], 1243–1253).—An apparatus for counter-current partition is described in which a series of cylindrical compartments are separated by plastic septa having a central hole 0·24 mm in diameter. A stationary phase is held in the cylinder while a mobile lighter or heavier phase is forced upwards or downwards respectively by compressed air. The pressure on the phase is raised sufficiently to atomise the droplets at the hole in the septum but is low enough to avoid emulsification and to allow the phase to clarify below (or above) the next septum. The mobile phase is collected after passage through the column of cylinders in an automatic fraction collector. The method is applied to partition of amino-acids between *n*-butanol and phosphate buffers at various pH, and the results obtained are compared with those of chromatography. The theory and uses of the apparatus are discussed. E. J. H. BIRCH

597. **Use of tangent intercept to determine an average speed of sedimentation.** W. P. Reid (*Anal. Chem.*, 1953, **25** [10], 1562–1563).—A method for the determination of average speed of sedimentation is described. G. P. COOK

598. **Three-stage molecular still.** D. A. Sutton (*Chem. & Ind.*, 1953, [52], 1383–1385).—A three-stage falling-film mol. still having ≃ 2·5 times the separation efficiency of a one-stage still is described and illustrated, and the method of operation is explained. The separate heated stages are mounted vertically above each other in a single tube 73 cm long, the distillate from the top stage being gravity-fed into and redistilled in the next lower stage, and so on; only one outlet to pump is thus needed. The residues from each stage are combined and circulated again by means of an air-lift pump of low hold-up. The efficiency was 1·25 theoretical plates for a three-stage distillation of a mixture of di-(2-ethylhexyl) phthalate and di-(2-ethylhexyl) sebacate containing 10 mol. per cent. of the former compound. The design of multi-stage stills is discussed. W. J. BAKER

599. **An inverted micro-osmometer and its use for the determination of molecular weight of some specimens of potassium hyaluronate.** J. A. Christiansen and C. E. Jensen (*Acta Chem. Scand.*, 1953, **7** [9], 1247–1254).—A dynamic equilibrium micro-osmometer is described and illustrated in which ≃ 500 μl of sample is contained in a semi-permeable cap fixed to the upper end of a vertical capillary. This is completely immersed in boiled 0·2 *M* KCl solution. The pressure on this outer liquid can be varied from approx. 0·1 to 1·0 atm., and the temperature is maintained at 20° C by an outer water-jacket. Observations of the length and movement of the air column in the capillary between the inner and outer liquids are made with a travelling microscope, and at each pressure difference the flow through the membrane in 10 min. is determined. Devices for filling the osmometer and for fixing a rubber ring to secure the semi-permeable cap are also described and illustrated. Results with solutions of K hyaluronate are of the same order as those reported previously by Jensen with similar solutions (*Acta Chem. Scand.*, 1953, **7**, 603). C. E. SEARLE

600. **Evaporation error in volume fractionation chromatography.** C. Mader and G. Mader, jun. (*Anal. Chem.*, 1953, **25** [10], 1556–1557).—A pipette switch arrangement, which is simple, inexpensive and easy to construct, is described. This arrangement eliminates evaporation error by saturation of the air that passes into the pipette switch when it empties and prevents evaporation of the eluting soln. Circuit diagrams are included. G. P. COOK

601. **Circular paper chromatography. Studies of physical factors that may influence R_F values.** A. Saifer and I. Oreskes (*Anal. Chem.*, 1953, **25** [10], 1539–1544).—Factors investigated include (*i*) effect of the presence of the inverse phase, (*ii*) effect of saturation of paper with solvent, (*iii*) effect of variation in length and width of paper wick, and (*iv*) variation of temp., time, concn. and ageing of solvent. Study of these factors showed that the method of Rutter (*Brit. Abstr. C*, 1949, 434; 1950,

215) when used in a closed system has many advantages as a paper chromatographic procedure. With a slightly modified system and phenol as developing solvent a mixture of 3 amino acids give R_F values with a standard deviation of $\pm$ 0·02 units or less. No significant changes in R_F values are produced by factors (*i*) and (*ii*), variation in width of wick, time, concn. and use of stored solvent. Factors influencing R_F values include temp. and length of exposed wick. Experimental procedure for the application of circular paper chromatography, including a diagram of the system, is given.
G. P. Cook

602. **A self-recording strip photometer for paper electrophoresis and paper chromatography** J. K. Miettinen and T. Moisio (*Acta Chem. Scand.*, 1953, **7** [9], 1225–1238).—Paper strips after electrophoresis or chromatography are passed through a filter photometer, and variations in absorption are recorded automatically by a polarograph galvanometer. An extinction scale is previously photographed on the sensitive paper. Duplicate determinations on a given strip vary by $< 0{\cdot}5$ per cent.; the accuracy of the method depends on the quality of the chromatograms. The application of the method to the electrophoretic analysis of human serum is described. C. E. Searle

603. **A modified all-glass apparatus for the determination of nitrogen by the micro-Kjeldahl method.** F. J. Scandrett (*Analyst*, 1953, **78**, 734–737).—In the all-glass apparatus described, an inlet for superheated steam, a funnel for introduction of NaOH soln. and an air condenser fit by means of a ground-glass joint into the neck of the Kjeldahl digestion flask. Distillation is by means of superheated steam and errors arising from transfer of the digest to another distillation flask are avoided. The apparatus could be used for the separation and determination of other volatile substances and, by replacement of the digestion flask by a larger flask with the same standard joint, could be used for macro-Kjeldahl N determinations. A. O. Jones

604. **An apparatus for dead-stop end-point titrations with acoustic indication of the end-point.** H. A. Glastonbury (*Analyst*, 1953, **78**, 682–683).—The apparatus described (with circuit diagram) provides an audible indication of approach to the end-point in dead-stop titrations. It is applicable to all such titrations (*e.g.*, titration of water with the Karl Fischer reagent) but not, without modification, to titrations with other electrode systems.
A. O. Jones

605. **Mortar and pestle with tungsten carbide surfaces.** R. S. Young (*Ind. Chem.*, 1953, **29**, 542).—A modified form of mortar and pestle fitted with tungsten carbide pulverising surfaces is described.
I. J. Metcalfe

606. **Soldering of Be, Al, Zr, U, W and Al-Li alloy.** E. Creutz (*Rev. Sci. Instrum.*, 1953, **24**, [4] 330–331).—The metal is "tinned" by dipping it through a molten flux into a molten solder of the appropriate composition described. Soldering is then accomplished by the standard methods. For Be and Zr the flux is Cu_2Cl_2. For Al, U and Al-Li alloy $ZnCl_2$ is used; for W borax is suitable flux.
G. Skirrow

Optical

607. **Examinations of surfaces by X-ray reflection.** R. H. Buteux (*J. Opt. Soc. Amer.*, 1953, **43** [7], 618).—Striations attributable to imperfection of surfaces observed in X-ray reflections have also been found to occur in mica and other glass-type materials, polished alkali halides and evaporated films of a large number of metals and non-metals. A nitrocellulose film coated with evaporated metals gave the appearance of a highly reflecting surface, although undulations were still observed. It is suggested that the difficulty in producing even surfaces is caused by elements of the surface film acting as curved reflectors, analogous to the Chinese and Japanese "magic mirrors." E. L. Seymour

608. **Analysis of fluorescent X-radiation by means of proportional counters.** R. E. Rowland (*J. Appl. Phys.*, 1953, **24** [6], 811–812).—A method is described for the rapid analysis of the X-ray spectrum from a fluorescent target irradiated by the beam from an X-ray tube. The fluorescent radiation is passed through a proportional counter filled with 90 per cent. A and 10 per cent. CH_4, at 1 atm., and the output of the tube is recorded with a motor-driven single-channel analyser; the results are registered on a Brown potentiometer. The counter is valuable in evaluating the energy and source of spurious X-rays. The purity of a Ti beam has been estimated as 95 per cent. with a self-rectified tube with tungsten anode and 1-mm beryllium window operated at peak voltage 9 kV. The purity decreases with increasing peak voltage. L. J. Jolley

609. **Absorption of β-rays by solutions, colloids and suspensions.** E. Canals, R. Marignan and L. Bardet (*Ann. Pharm. Franç.*, 1953, **11** [9–10], 588–594).—An apparatus is described in which 2·3-MeV β-rays from a UO_2 source are passed through a cell with a mica base (30 mg/sq. cm), between Pb collimating screens to a Geiger-Müller counter. The absorption coeff., μ, and the index, n, in the expression $I = I_0e^{-\mu x^n}$, where x is the thickness of solution in the cell, is determined for a number of solutions and colloids including blood and serum. The thickness of the solution required to reduce the intensity to 1/1000 of the incident radiation is $\simeq$ 10 times that to reduce it by half.
E. J. H. Birch

610. **Semi-automatic recording of wavelength data.** G. K. Werner (*J. Opt. Soc. Amer.*, 1953, **43** [7], 620–621).—An instrument is described whose basic components are a digital converter with a manually-controlled variable speed motor, a 100 to 1 worm gear reduction coupling from digital converter to comparator, a Gaertner model M1205C comparator and an IBM numeric key punch machine for manual entry of information. The instrument is operated by aligning the spectrum line with the cross hairs of the comparator and pushing a button; the six-figure comparator setting is then automatically punched on an IBM card.
E. L. Seymour

611. **Vacuum-tube molecular emission and dissociation spectra.** J. H. Lee (*J. Opt. Soc. Amer.*, 1953, **43** [7], 619).—The results obtained from spectra excited in a glow discharge by electronic techniques (Talley, Lowe and Scanion, *J. Opt. Soc. Amer.*, 1952, **42**, 982) and those obtained earlier (Coblenz, "Investigations of Infra-red Spectra," Carnegie Institution of Washington Publication, No. 35, 1905) are compared and discussed. Dissociation phenomena, the elimination of the continuum from the discharge tube and the failure of Coblenz to observe certain emission bands of CO_2 and H_2O are reviewed. E. L. Seymour

612. **Emission spectroscopic methods with special reference to the determination of calcium in biological research material. A collective review.** H.-U. Riethmüller (*Mikrochim. Acta*, 1953, [3], 178–195).—Papers concerned with the emission spectroscopic determination of Ca either alone or in the presence of other elements in all types of material are summarised in tabular form. A table is included showing the advantages and disadvantages of the various methods of exciting spectra. Because of their speed, accuracy, relative simplicity and cheapness, flame photometric methods are most frequently used. Papers dealing with the method of use of various types of flame photometer, particularly for determining Na, K and Ca, are also summarised. J. H. WATON

613. **Carbon arc as an infra-red source.** J. H. Jaffe (*J. Opt. Soc. Amer.*, 1953, **43** [7], 619).—The disadvantages of the large dimensions of the carbon arc source of Rupert and Strong (*J. Opt. Soc. Amer.*, 1950, **40**, 455) can be avoided by the construction of small electrodes in conjunction with a constant current circuit. Performance of such a source is similar to that reported by Strong and is also able to replace the Globar source. E. L. SEYMOUR

614. **Reproducibility of measurements made on a Cary spectrophotometer and its log-density attachment.** J. L. Forstner and L. B. Rogers (*Anal. Chem.*, 1953, **25** [10], 1560–1562).—The points examined are (*i*) accuracy of slide wire on the log-density recorder, (*ii*) reproducibility of the absorbance readings obtained at different rates of scanning the λ scale, (*iii*) effect of slit adjustment on the absorbance values, (*iv*) errors resulting from failure of slit to change sufficiently fast when highly absorbant reference solutions are used. Conclusions reached are that (*i*) is accurate over the absorbance range of 0·1 to 1·85, reproducibility becomes a little poorer as the scan rate increases, variation of slit control between 10 and 50 does not produce much change in the spectrum but below 10 the change is rapid, and the slit mechanism is able to cope with scan rates of 5 A per sec.; results fall off as much as 20 per cent. at 20 A per sec. G. P. COOK

615. **Compensation of astigmatic errors in a grating spectrograph.** H. W. Straat (*J. Opt. Soc. Amer.*, 1953, **43** [7], 593–594).—The principles involved in the correction of simple types of grating spectrographs exhibiting astigmatism as a function of λ are outlined. The most advantageous correction system is found in form of a simple cylindrical lens between a concave grating and the slit. Examples of corrected spectra demonstrate a high order of image perfection by correct matching of grating and lens. E. L. SEYMOUR

616. **A portable fluoroscope using ultraviolet from daylight.** J. W. Walley (*Chem. & Ind.*, 1953, [48], 1278–1279).—A small portable non-electric fluoroscope is described. The u.v. light required for the irradiation of specimens is filtered from sunlight. The fluoroscope (illustrated) consists of a sheet steel box (5 × 5 × 5 in.) with a hinged window (3 × 3 × 0·2 in.) of Wood's glass at the top and an eye-piece tube entering the front side obliquely. The window functions as a combined u.v. filter and insertion door for specimens, whilst the inspection tube (with a rubber eyeguard) is adjustable to maintain the specimen in focus. A turntable in the base of the box permits rotation of the specimen whilst viewing. Excellent results are obtained with a wide variety of fluorescent materials even on a dull day. Applications of the instrument are discussed and future developments outlined. A less sensitive and more fragile model also is described consisting of a Wood's glass flask with a removable brass tray for insertion and ex action of specimens. D. BAILEY

617. **Monolayer fluorescent screens.** L. R. Koller (*J. Opt. Soc. Amer.*, 1953, **43** [7], 620).—A process is described for the production of a single-layer screen of closely packed phosphor grains. The grains are treated with silicone, a water repellent, the particles are then dusted on a clean H_2O surface, and the film is compressed on to a glass surface. The performance of such a screen is found to be satisfactory and the process is recommended for the preparation of fluorescent screens of higher quality and smaller thickness. E. L. SEYMOUR

618. **Radiometer calibration for extended-source measurements.** F. E. Nicodemus (*J. Opt. Soc. Amer.*, 1953, **43** [7], 547–549).—Brightness or steradiance of an extended source is measured with a photometer or radiometer with non-uniform directional response. The calibration method used involves the establishment of a relative calibration scale of radiation energy, its transformation into absolute units of radiancy from a distant point source, and finally its conversion into units of brightness or steradiance of the extended source. Detail of the individual steps in the calibration and a diagram of the apparatus for extended-source calibration are given. E. L. SEYMOUR

619. **Photo-electric cells for the vacuum ultra-violet.** H. E. Hinteregger and K. Watanabe (*J. Opt. Soc. Amer.*, 1953, **43** [7], 604–608).—The photo-electric effect on Ni, Pt and W has been utilised in the design of windowless phototubes for the study of radiation below the transmission limit of LiF. An extremely high quantum yield (10 photo-electrons per 100 incident quanta) has been observed for these metals, so permitting the tubes to be operated with the same electronic instruments and automatic scanning down to negative voltages. Details of the photocell, the circuit and the spectral characteristics are given. Results show good reproducibility of response after exposure to air and adjustability of the response by the external circuit. Some of the applications and possible further development of the short-wave detectors are outlined. E. L. SEYMOUR

620. **Automatic calculation of colour differences.** A. Opler, R. W. Meikle and M. J. Charlesworth (*J. Opt. Soc. Amer.*, 1953, **43** [7], 550–551).—A routine procedure for the calculation of colour differences has been developed to enable several thousands of such calculations to be made by an entirely mechanised procedure by means of punched-card operated computing machines, which convert the C.I.E. tristimulus colour space into the equal visual stimulus space of Adam. E. L. SEYMOUR

621. **An interferometer for simultaneous observation of concentration and concentration gradient in liquid columns.** W. Weinstein (*Nature*, 1953, **172**, 461–462).—An optical system comprises a point source of light, polarising screen, Wollaston prism, collimator and plane mirror; two coherent plane-polarised wave-fronts are formed which pass through two cells situated side by side, one containing the liquid of varying concn. and the other homogeneous

liquid. Interference fringes are formed, which can be photographed; in one region they map the concn. and in another map the concn. gradient throughout the cell. H. P. PAGET

Thermal

622. **A meniscus thermometer for the measurement of small temperature differentials.** S. J. Borgars (*J. Sci. Instrum.*, 1953, **30** [12], 487).—A thin-walled glass bulb is blown on the end of a length of capillary tubing. A short column of liquid in the capillary indicates, from its change of position, temp. changes of the bulb. Corrections for changes in barometric pressures are necessary, but temp. differences may be measured to within ± 0·004°. G. SKIRROW

623. **A fast, electro-optical, hot-gas pyrometer.** G. H. Millar, J. G. Winans, O. A. Uyehara and P. S. Myers (*J. Opt. Soc. Amer.*, 1953, **43** [7], 609–617).—A detailed description is given of a fast electro-optical pyrometer that records temp. of flames naturally luminous, including the sodium D-lines, or flames made luminous by Na additions. The optical design, particularly lens relationship, stop position and conditions of optimum slit-length diaphragm-opening combinations, is discussed. The validity of the emissivity and absorptivity is critically examined. E. L. SEYMOUR

624. **Burning time and ignition temperature apparatus for metal powders.** H. C. Andersen and L. H. Belz (*Rev. Sci. Instrum.*, 1953, **24** [10], 1004).—A photo-electric apparatus is used for determining the time taken for a flame to travel along 10 in. of a track ⅛ in. wide by 1/16 in. deep, filled with the metal powder. The ignition temp. is measured by heating a sample of the powder on a Cu block and observing the temp. at which the first glow appears. G. SKIRROW

625. **Preparation and some properties of conducting transparent glass.** R. Gomer (*Rev. Sci. Instrum.*, 1953, **24** [10], 993).—A conducting coating can be deposited on a glass object by heating the object in an oven to 400° C and blowing over it the vapour from heated $SnCl_2.2H_2O$. Such coatings are transparent for resistivities of approx. 0·025 ohm-cm. High conductivity coatings (10^{-3} ohm-cm) may be milky or light blue. All coatings are resistant to boiling HNO_3, to heat and to abrasion. It is suggested that some specimens may be useful as resistance thermometers at liq. helium temperatures. G. SKIRROW

Electrical

626. **Renewable, constant-area mercury cathode [for polarography].** M.-P. Simonnin and M. Quintin (*Compt. Rend.*, 1953, **237** [22], 1409–1411).—In the arrangement shown the cathode is formed by a column of Hg in a thin tube, renewal being effected by drops of Hg from a capillary immediately above. Reproducible and normal polarograms are obtained by applying variable voltage between the cathode and, *e.g.*, an Ag - AgCl electrode immersed in a soln. such as 0·1 *N* KCl plus 0·0013 *M* $CdCl_2$. By passing pure N continuously through the solution, the O wave is suppressed without the need for adding a reducing agent or other suppressor. W. J. BAKER

627. **Polarographic analysis. [Application in petroleum industry.]** A. Poussin (*Rev. Inst. Franç. Pétrole*, 1953, **8**, 504–512).—A review with 36 references. A. R. PEARSON

628. **Properties of the antimony micro-electrode in aqueous alcoholic solutions.** B. Kamieński, Z. Bylo and B. Waligóta (*Bull. Acad. Polon. Sci.*, 1953, **1** [3–4], 137–141).—Graphs are shown that indicate the approx. linear increase in negative potential of Kamieński's modification of the Sb micro-electrode (*vs.* a 0·1 *N* KCl - Hg_2Cl_2 electrode) with decrease in the $[H^+]$ for various concn. of aq. soln. of methanol, ethanol or propanol. From these it follows that small changes in composition of binary alcohol - water mixtures will not impair accuracy in potentiometric adsorption analysis. The effect of small additions of *n*-butyric, *n*-valeric, *n*-hexoic, *n*-heptoic, *n*-octoic or *n*-myristic acid to the aq. methanolic soln. and of cinchonine or brucine to the aq. ethanolic soln. are also illustrated. B. J. W.

629. **Purity tests with the help of dielectrometric analysis.** G. Ebert (*Z. anal. Chem.*, 1953, **140** [3], 161–166).—The purity of several chemical substances is determined by using their dielectric constant ϵ as a criterion of purity. The examined substance is subjected to fractional distillation and the ϵ of the distillate is measured continuously in a cell connected to a cooler. The constants ϵ are determined at 20° C using ν of 7050 kilocycles per sec.; the accuracy of the method is ± 0·3 per cent. Re-determination of the ϵ of the same org. components and comparison with the values reported in literature is undertaken. S. K. LACHOWICZ

630. **An apparatus for the measurement of the thermal conductivity of biological tissue.** H. S. Hatfield (*J. Sci. Instrum.*, 1953, **30** [12], 460–461).—The sample in the form of a disc 12 mm in diam. and 3 mm thick is sandwiched between two disc-shaped Te - Ag thermocouples. The lower thermocouple is heated and conditions are adjusted so that the two heat-flow meter discs are brought to the same reading. The temp. gradient through the discs and specimen is measured by thermocouples. A blank determination with no specimen between the discs enables the correction due to the thermal resistance of the discs to be calculated. G. SKIRROW

631. **Diffusion of electrolytes and the polarographic method. The limiting current of a reducible ion on the dropping-mercury cathode.** Ya. P. Gokhshteyn (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [2], 71–83).—The validity of the diffraction method for measuring coeff. of diffusion is demonstrated. Tabulated data illustrate the effect of base electrolyte concn. on the true diffusion const. The limiting current of a reducible ion in presence of a large excess of base electrolyte on the dropping Hg cathode consists in the diffusion current and an additional current. The additional current, not considered in the Ilkovič equation, is on the average ≃ 23 per cent. of the main radial diffusion current when capillaries of drop times 2 to 7 sec. are used. Experimental data show that the Ilkovič equation requires modification to $I_{11m.} = 744{\cdot}2\ zcD^{\frac{1}{2}}m^{\frac{2}{3}}t^{\frac{1}{6}}$ for reduction of cations and anions of the type $HPbO_3'$, and to $I_{11m.} = 465{\cdot}9\ zcD^{\frac{1}{2}}m^{\frac{2}{3}}t^{\frac{1}{6}}$ for anions of the type IO_3' and BrO_3'. The equation of Strehlow and Stackelberg (*Z. Elektrochem.*, 1950, **54**, 51) does not accord with the experimental data. G. S. SMITH

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.	micro-litre	μl
ampere	amp.	micron	μ
Ångstrom unit	A	milliampere	mA
anhydrous	anhyd.	milligram	mg
approximate, -ly	approx.	millilitre	ml
aqueous	aq.	millimetre	mm
atmospher-e, -ic	atm.	millivolt	mV
atomic	at.	minimum	min.
boiling-point	b.p.	minute (time)	min.
British thermal unit	B.Th.U.	molar (concentration)	*M*
calculated	(calc.)	molecul -e, -ar	mol.
calorie (large)	kg-cal.	normal (concentration)	*N*
calorie (small)	g-cal.	number	no.
centimetre	cm	observed	(obs.)
coefficient	coeff.	organic	org.
concentrated	conc.	ounce	oz.
concentration	concn.	part	pt.
constant	const.	patent	pat.
corrected	(corr.)	parts per million	p.p.m.
critical	crit.	per cent. wt. in wt.	per cent. w/w
crystalline, crystallised	cryst.	per cent. wt. in vol.	per cent. w/v
		per cent. vol. in vol.	per cent. v/v
cubic	cu.	potential difference	p.d.
current density	c.d.	pound	lb
cycles per second	c.p.s.	precipitate	ppt.
decompos -ing, -ition	(decomp.)	precipitated	pptd.
density	ρ	precipitating	pptg.
density, relative	*d* or wt. per ml	precipitation	pptn.
derivative	deriv.	preparation	prep.
dilute	dil.	qualitative, -ly	qual.
direct current	d.c.	quantitative, -ly	quant.
distilled	dist.	recrystallised	recryst.
electromotive force	e.m.f.	refractive index	$n\lambda^t$
electron-volt	eV	relative humidity	R.H.
equivalent	equiv.	revolutions per minute	r.p.m.
experiment, -al	expt.	saponification value	sap. val.
gram	g	saturated calomel electrode	S.C.E.
gram-molecule	mole	second (time)	sec.
half-wave potential	$E_{\frac{1}{2}}$	soluble	sol.
horse-power	h.p.	solution	soln.
hour	hr.	specific gravity	sp. gr.
hydrogen ion concentration	[H·]	specific rotation	$[\alpha]\lambda^t$
hydrogen ion exponent	pH	square centimetre	sq. cm
inch	in.	standard temperature and pressure	s.t.p.
indefinite	indef.	temperature	temp.
infra-red	i.r.	ultra-violet	u.v.
insoluble	insol.	vapour density	v.d.
kilogram	kg	vapour pressure	v.p.
kilovolt	kV	volt	V
kilowatt	kW	volume	vol.
liquid	liq.	watt	W
maxim -um, -a	max.	wavelength	λ
melting-point	m.p.	weight	wt.
microgram	μg		

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

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